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ATTACHMENT 2
CHESTER LabNet
Laboratory SOP

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TABLES

1.0 INTRODUCTION

The Analytical Division of Keystone Environmental Resources, Inc., is committed to excellence in chemical analysis. All data generated by the Division must be technically sound, properly documented, legally defensible, and supported by defined and verified confidence limits.

This document is designed to serve as a guideline to the Division as a whole. Specifically, this document defines the divisional objectives, organization, functional activities, and QA/QC programs that routinely apply to the entire Division. This document is supplemented by sets of Standard Operating Procedures that are unique to the separate laboratories. This document delineates the standard practices within Keystone Analytical Division; for specific projects, addenda will be prepared responding to the project needs.

2.0 OBJECTIVES

The Quality Assurance Program at Keystone Analytical Division is principally aimed at producing results of verifiable high quality. Towards this goal, the Program addresses several areas:

1. Detection of problems through statistical measures of acceptability and confidence

2. Implementation of corrective action

3. Documentation procedures designed to produce legally defensible results

4. Establishment of training programs to assure that each person is thoroughly familiar with the methods, procedures, and documentation of his area of activity

5. Development of a review and validation process to verify that all data produced by the Division are within the guidelines defined in this Manual and the associated Standard Operating Procedures.

3.0 ORGANIZATION AND RESPONSIBILITY

Before delving into the the procedural aspects of this Manual, it is necessary to define the organizational structure of the Division so that reporting and responsibility levels can be outlined.

3.1 DIVISION

3.1.1 Organization

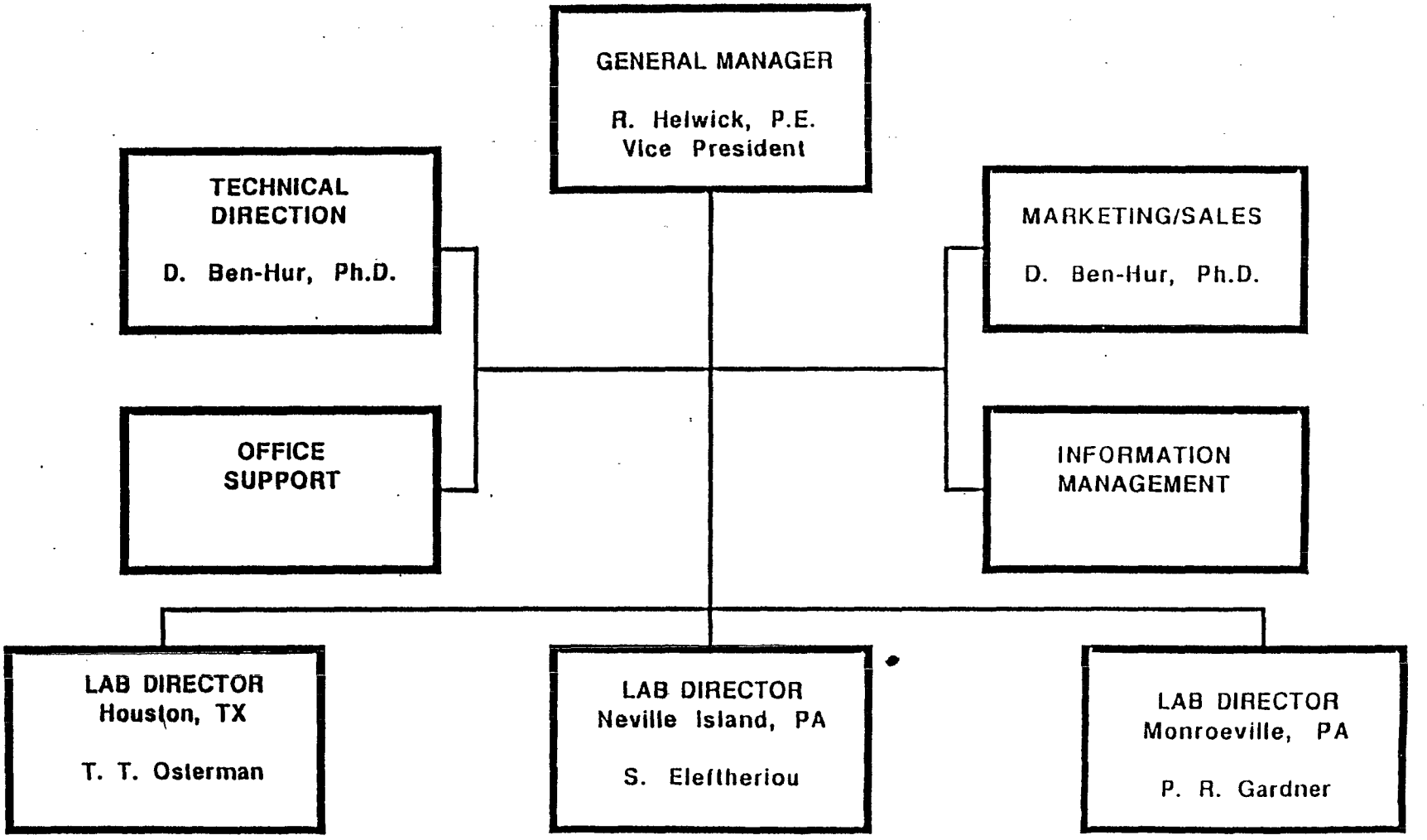
The Divisional organization is schematically shown in Figure 3-1.

3.1.2 Responsibility

While the functions of Divisional personnel are principally administrative, certain responsibilities relative to the Quality Assurance Plan do exist.

The Division **Operations Manager** is responsible for assuring that the Laboratory Directors, the Technical Director, and the Laboratory Information System Manager are thoroughly familiar with the Divisional Quality Assurance Manual and good laboratory practices. It is also the responsibility of the Operations Manager or his designee to approve all changes and revisions in the Divisional Quality Assurance Manual.

The **Information System Manager** is responsible for assuring that all reports generated by the system are correct and complete, and for verifying that all necessary back-up data are available. It should be emphasized that reports are generated at two different levels. For projects that are entirely internal to a single Laboratory, the reports are generated, approved and verified in that Laboratory. Reports on projects that involve several laboratories require integration of data. The assignment of the responsible laboratory, and coordination of the integration of data for reports involving several laboratories is the responsibility of the Information System Manager. Coordination and distribution of samples on projects that necessitate employing more than one facility will also be performed by the Information System Manager.



DIVISION ORGANIZATION CHART

The **Technical Director** serves the role of establishing appropriate protocols within the individual laboratories and assuring that the Division quality assurance program is being followed. In cases of deviations from established procedures, the approval of the Technical Director must be obtained. The Technical Director will also review and approve all new methodologies established in any of the laboratories. It is the responsibility of the Technical Director to assure that all work performed by the Division meets technically sound criteria.

The role of the Laboratory Directors will be discussed in the next Section.

3.2 LABORATORY

3.2.1 Organization

Figure 3-2 illustrates generically the organization of each Laboratory within the Division.

3.2.2 Responsibility

The **Laboratory Director** is responsible for assuring that all Section Managers are thoroughly familiar with the Divisional Quality Assurance Manual and good laboratory practices, and that all laboratory personnel meet the requisite qualifications for their positions within the laboratory. The Laboratory Director, or his designee, must review and approve all outgoing reports. The Laboratory Director is also responsible for effective daily management of the laboratory and its staff, and for communication and liaison with the client.

The **Section Manager** is responsible for the production of quality results within the Section. To achieve this, the Section Manager must be thoroughly familiar with the Divisional Quality Assurance Manual and the associated Standard Operating Procedures for his Section. He is also responsible for familiarizing the Section personnel with the Quality Assurance Manual and the

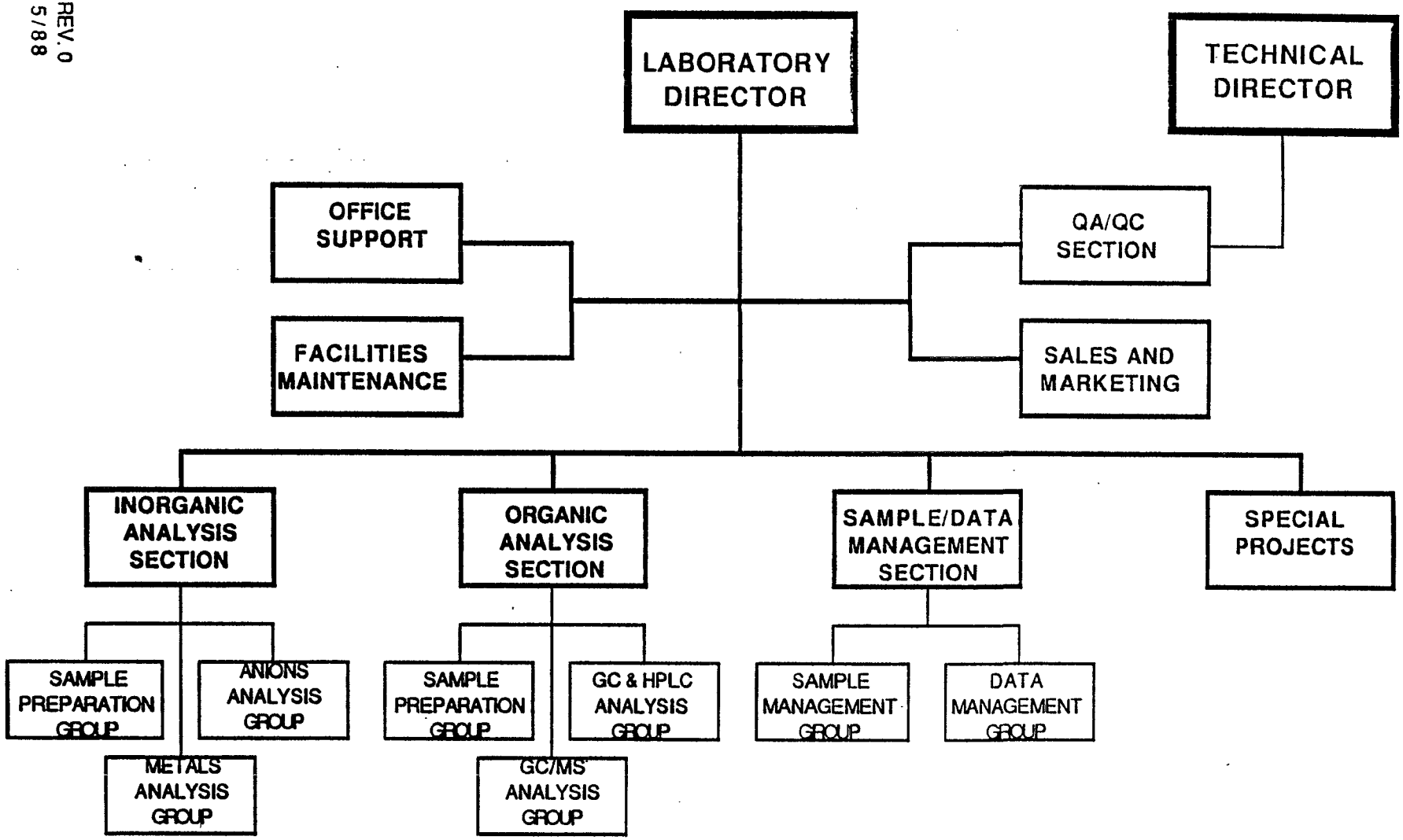


FIGURE 3-2 LABORATORY ORGANIZATION CHART

Standard Operating Procedures, overseeing that required protocols are followed, reviewing the results, and approving release of the data to the Data Management Group. The Section Manager, in coordination with the Sample and Data Management Section and the Project Manager, if a special project is involved, is responsible for the scheduling of work in the Section and conforming to required holding times. The Section Manager, in conjunction with the Quality Assurance Manager, is responsible for providing the necessary training to the Section personnel.

The **Group Leaders** and **analysts** in the Inorganic Section, the Organic Section, and in Special Projects are responsible for performing all analyses as required, paying attention to the required QC analyses demanded by the analytical method or technique. In order to provide proper analysis, they must be familiar with the Quality Assurance Manual and the associated Standard Operating Procedures. They are also responsible for initiating system or method corrective action, should they become aware of a malfunction. Initiation of corrective action requires appropriate notification, as discussed later in this Manual.

The **Sample and Data Management Section Manager** is responsible for the coordination of the activities of the Sample Management Group and the Data Management Group. It is his responsibility to assure that the group leaders are thoroughly familiar with the Quality Assurance Manual. He is also responsible for interacting with clients in case of discrepancies or irregularities in the sample shipment. In addition, he is responsible for maintaining and updating the schedules within the Department.

The **Sample Management Group Leader** is responsible for the following functions: sample receipt, storage, distribution of the information through the Laboratory, sample custody, and sample disposal. It is his responsibility to notify the Section Manager should there be any discrepancies or irregularities in the shipment of samples.

The **Data Management Group Leader** is responsible for maintaining the status of the work within the Laboratory, coordinating the compilation of the data, and preparation of reports for review and approval. It is also his

responsibility to alert the Section Manager if there are apparent problems in meeting schedules or holding times.

The **QA/QC Manager** is responsible for assuring that the QA/QC requirements of the Quality Assurance Manual and its associated Standard Operating Procedures and addenda are strictly adhered. He is responsible for review and validation of data, alerting the Sections should the need for corrective action exist, performing internal audits as specified by the Manual, introduction of performance evaluation samples on a periodic basis, and maintenance of the QC records. He is also responsible for preparing project specific QA/QC plans, and interfacing with clients on matters pertaining to data quality.

The QA/QC Manager functions independently of the laboratory staff. In order to achieve independence from the pressures of daily production in the laboratory and maintain the necessary objectivity, the QA/QC Manager reports both to the Laboratory Director and the Divisional Technical Director.

The **Project Manager** is a special position assigned by the Laboratory Director for specific projects. Projects may require a specifically assigned manager because of unusual duration of the project, complexity of the analytical techniques or reporting requirements, and coordination of activities in several laboratories. The responsibility of the Project Manager to the specific project transcends that of the Laboratory Director. It is the Project Manager's responsibility to assure that work on the project is performed in accordance with project specified protocols, following project specific QC requirements. Acceptance of results on analyses for the project is subject to approval by the Project Manager.

4.0 SAMPLE CUSTODY

To provide for legal defensibility of all work performed at a given site, it is essential to be able to provide documentation tracing the samples from collection, to the laboratory, and through the analytical procedures. Keystone performs both sampling functions and analytical functions; however, the Division can only guarantee that this Manual is followed from the point of origin only for samples that are both collected and analyzed by Keystone.

Maintaining sample custody consists of two distinct aspects: maintenance of the samples in the field, and maintenance of the samples from the time of receipt in the laboratory. These two aspects are discussed separately in the following sections. Inasmuch as sampling is not necessarily performed by our personnel, the custody and documentation in the field are included here as a recommendation.

4.1 CUSTODY AND DOCUMENTATION IN THE FIELD

The field sample custodian, which, depending upon the project, may be the sampler or another person in the same sampling group is considered to have custody of the samples at all time during the field operations, until the samples are shipped to storage or to the laboratory. Upon collection of a sample in the field, the sampler tags the sample with its site and type (water, soil, sludge, etc.) identification. The sampler also indicates on the tag the date and time of sampling. After cleaning the exterior of the sample container, the field sampler transfers the container with the tag to the field sample custodian.

Throughout this document, the term sample is used to indicate a quantity of one type of material collected at one time, at a single location. Thus, a water sample may be shipped to the laboratory in several containers, depending upon the required testing and sample preservation dictated by the project. Each container is identified as a sub-sample, but it is not classified as a unique sample.

The field sample custodian compares the identification of the individual sample with the sampling plan, and enters all pertinent information on the chain-of-custody document and on the label of the sub-sample container. The information that must be included consists of the following:

a. Project identification
b. Sample identification (such as station number and location)
c. Date of sampling
d. Time of sampling
e. Name of sampler
f. Parameters for which the sample is to be analyzed
g. Number of containers
h. Sample matrix (ground water, surface water, wastewater, soil, sediment, sludge, unknown waste, etc.)
i. Added preservatives in each sample container
j. Ice chest number
k. Chain of custody number

A sample field chain-of-custody is shown in Figure 4-1.

The field sample custodian is then responsible for packaging the sample(s) for shipping, adding ice if the samples require chilling, signing and dating the chain of custody document, placing the chain of custody document in a water-proof envelope and attaching the envelope to the inside of the ice chest lid.

If the samples are to be shipped by a common carrier, then the field sample custodian must also place custody seal on the ice chest.

Simultaneously with filling the chain of custody document, the field sample custodian also records the information in the field logbook. In filling the chain of custody document and the field logbook, any corrections that need to be made must be done so that the original incorrect entry is legible. Hence, the incorrect entry is lined out, and the change is initialed and dated by the field sample custodian.

Table 4-1 lists the required types of containers, preservatives, and holding times for each type of analyte. It is the responsibility of the field sample custodian to assure that each sample or sub-sample are packaged correctly. The samples are considered formally to be in the custody of the field sample custodian until they are officially transferred to the carrier, and the transfer is documented on shipping records, or until the samples are transferred to the laboratory in person.

FIGURE 4-1

CHAIN OF CUSTODY RECORD

PLANT CODE	PROJECT NAME	NUMBER OF CONTAINERS
SAMPLERS *(Signature)*		

STA NO.	DATE	TIME	COMP	GRAB	WELL	STATION LOCATION	CONDUCTIVITY	pH	REMARKS OR OBSERVATIONS

Relinquished by: *(Signature)*	Date	Time	Received by: *(Signature)*	Reliquished by: *(Signature)*	Date	Time	Received by *(Signature)*
Relinquished by: *(Signature)*	Date	Time	Received by: *(Signature)*	Reliquished by: *(Signature)*	Date	Time	Received by *(Signature)*
Reliquished by: *(Signature)*	Date	Time	Received for Laboratory by: *(Signature)*	Date	Time	Ice Chest Temp ^{o}C	Ice Chest #

Chain of Custody Tag #

*DISTRIBUTION Original accompanies shipment; Copy to Coordinator Field Files.

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TABLE 4-1
CONTAINERS, PRESERVATIVES, AND HOLDING TIMES

ANALYSIS	CONTAINER[1]	PRESERVATIVE	HOLDING TIME[2]
Volatile organics	G	Cool to 4°C	14 d
Semivolatile organics	G	Cool to 4°C	7 d to extraction, 40 d for extract.
Organochlorine pesticides	G	Cool to 4°C	7 d to extraction, 40 d for extract.
Herbicides	G	Cool to 4°C	7 d to extraction, 40 d for extract
Organophsophorus pesticides	G	Cool to 4°C	7 d to extraction, 40 d for extract
Carbamates	G	Cool to 4°C	7 d to extraction, 40 d for extract
Triazines	G	Cool to 4°C	7 d to extraction, 40 d for extract
Metals (except mercury)	P	HNO_3	6 m
Mercury	P	HNO_3	28 d
Hexavalent chromium	P	Cool to 4°C	24 h
Acidity, Alkalinity	P, G	Cool to 4°C	14 d
Ammonia, COD, total phosphorus	P, G	H_2SO_4, Cool to 4°C	28 d
BOD	P, G	Cool to 4°C	48 h
Chloride, Fluoride	P	None	28 d
Color	P, G	Cool to 4°C	48 h
Cyanide	P, G	NaOH, Cool to 4°C	14 d
Hardness	P	HNO_3	6 m
pH	P, G	None	None

TABLE 4-1
CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
(CONTINUED)

ANALYSIS	CONTAINER[1]	PRESERVATIVE	HOLDING TIME[2]
Nitrogen, Kjeldahl and Total organic, Phenols	G	H_2SO_4, Cool to 4°C	28 d
Nitrate, Nitrite	P, G	H_2SO_4, Cool to 4°C	48 h
Oil and Grease	G	H_2SO_4, Cool to 4°C	28 d
Ortho-phosphate	P, G	Cool to 4°C	48 h
Dissolved oxygen	G	None	None
Residue, Total, Filterable, Nonfilterable and Volatile	P, G	Cool to 4°C	7 d
Residue, Settleable	P, G	Cool to 4°C	48 h
Silica	P	Cool to 4°C	28 d
Sulfate, Specific Conductance	P, G	Cool to 4°C	28 d
Sulfide	P, G	Zinc acetate + NaOH, Cool to 4°C	7 d
Sulfite	P, G	None	None
Surfactants, Turbidity	P, G	Cool to 4°C	48 h
Temperature	P, G	None	None

1) P = Polyethylene
G = Glass

2) h = hours
d = days
m = months
None means that analysis must be done immediately

4.2 SAMPLE CUSTODY IN THE LABORATORY

The laboratory operation, as it pertains to the sample custody, consists of several functions. Specifically, these are: sample receipt, inspection of the samples, reconciliation of the information on the sample label and the chain-of-custody, alerting the project manager or the Sample and Data Management Section Manager of any inconsistencies in the shipment, logging in of the samples, placing the samples in appropriate storage areas, distribution of the information to the laboratory analytical sections, transferring of the custody of the samples to the analysts, recovery of the samples at the completion of the analysis, and discarding of the samples after the appropriate laboratory holding time has expired.

These operations are the responsibility of the Sample Management Group. The precise steps are itemized below.

4.2.1 Sample Receipt in the Laboratory

Samples will be received in the laboratory either by commercial carrier, the postal service, or hand-carried. Personnel of the Sample Management Group sign for the receipt of each shipment of samples, and retain a copy of the shipping documents. The personnel receiving the sample shipment will open a sample shipment checklist, a copy of which is shown in Figure 4-2, at the time of receiving the shipment.

If, for any reason, the shipping container is not expected to be opened immediately, then the seals on the container must remain intact.

4.2.2 Shipment Inspection

It is expected that a shipment received in the laboratory will be opened and inspected immediately upon receipt. Prior to opening the shipping container, the custody seals will be inspected to assure that no tampering has been done with the sample containers. The state of the custody seals will be noted by the person inspecting the shipment of the sample shipment checklist.

KEYSTONE

☐ Houston ☐ Monroeville ☐ Ontario

sample shipment checklist

CLIENT: _____ DATE SHIPPED: _____

CLIENT CONTACT: _____ DATE RECEIVED: _____

TELEPHONE NO.: _____ SHIPPED VIA: _____

CHECKED BY: _____

NUMBER OF SHIPPING CONTAINERS (COOLERS, BOXES, ETC.): _____

CONTAINER ID	CUSTODY TAPE		TEMPERATURE C	NO. OF SAMPLE CONTAINERS	AGREE WITH COC? (Y/N)
	PRESENT? (Y/N)	INTACT? (Y/N)			

IRREGULARITIES

SAMPLE ID	SUB SAMPLE ID	IRREGULARITY

CHECKER SIGNATURE: _____ DATE: _____

RESOLUTION OF IRREGULARITIES WITH CLIENT

CLIENT REPRESENTATIVE: _____ KEYSTONE REPRESENTATIVE: _____

TELEPHONE NO.: _____ DATE _____ TIME _____

WRITTEN FOLLOWUP (Y/N) _____ DATE _____

DECISION: _____

_____

_____

_____

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SIGNATURE _____

FIGURE 4-2
SAMPLE SHIPMENT CHECKLIST

After the seals are inspected, the ice chest is placed in a fume hood and opened. The temperature of the interior of the ice chest is measured and recorded. To measure the temperature, the lid is quickly opened and a thermometer is inserted into the ice chest, and the lid is closed again for five minutes. At the end of five minutes, the lid is quickly opened and the temperature read rapidly. The temperature is recorded on the sample shipment checklist.

While the ice chest is still in the fume hood, the chain of custody document is removed from the inside of the lid, and the individual sample containers are removed from the ice chest. The sample or sub-sample containers are counted and reconciled with the number of such containers indicated on the chain-of custody. If the number of retrieved sample containers is less than that indicated on the chain-of-custody, the packing materials inside the ice chest are further checked to make sure that no sample container has been accidentally left in the ice chest.

Each individual sample or sub-sample container is visually inspected to determine that no breakage, cracking, external corrosion, or leakage has occurred. If none have occurred, the individual sample containers may be removed from the fume hood and placed on a workbench to complete the inspection. If, on the other hand, there is indication that breakage, cracking, corrosion, or leakage has occurred, the inspection of the sample containers will be completed while the containers are kept in the fume hood.

The integrity of the individual sample or sub-sample containers is recorded on the sample shipment checklist.

4.2.3 Reconciliation with Chain-of-Custody Document

Once the integrity of the sample containers has been determined, the sample containers are reconciled against the records on the chain-of-custody. This is done by checking the sample identification on the chain-of-custody and on the sample container label. In addition, the analyte identifications are checked to ensure that they are correct.

Any discrepancy is noted on the chain-of-custody, signed, and dated. The discrepancies are also entered on the sample shipment checklist. At this point, the sample shipment inspection is complete. The sample shipment checklist is signed and dated by the person performing the inspection. If there are no discrepancies, and the shipment is complete as evidenced by the inspection, the shipment of samples is ready to be logged in. If there are discrepancies or inconsistencies, the sample shipment checklist is submitted to the Manager of the Sample and Data Management Section, or his designee, and the logging in process is delayed until the discrepancies are resolved.

4.2.4 Resolution of Shipment Irregularities

If any irregularities are noted during the sample shipment inspection, they are recorded on the sample shipment checklist, and the checklist is submitted to the Manager of the Sample and Data Management Section or his designee. The Manager of the Sample and Data Management Section, or his designee, will contact the client's representative to determine the fate of the sample shipment. The records of the conversation with the client's representative are entered on the sample shipment checklist, including name of contact, time and date of the conversation, and the resolution of the irregularities.

There are several possibilities for the resolution of the irregularities. These are:

1. Return the sample shipment to sender
2. Destroy the entire shipment of samples
3. Log in and process those samples that are intact.

The sample shipment checklist containing the comments regarding resolution of any irregularities is returned to the Sample Management Group, the personnel of which will act according to the annotated agreement with the client.

To maintain the custody of the samples, the shipment of samples during this period is either locked up in a secure area or is in view of the Sample Management Group personnel.

4.2.5 Sample Log In

Once the sample shipment has been inspected and any irregularities resolved, the sample shipment is ready to be logged in. For laboratory purposes, a single sample shipment from a specific client constitutes a single job. The shipment may contain one or many samples, and may have arrived in a single shipping container or in many shipping containers.

For the log-in process, the Sample Management Group person performing the logging in needs the field chain-of-custody and the sample shipment checklist, both with whatever corrections required to be made during the inspection and resolution steps. The log-in process consists of four steps which are detailed below:

4.2.5.1 Entry in Master Log

The Master Log is a hardbound book in which all jobs received in the laboratory are chronologically recorded. The following information is entered in the Master Log:

1. Job Number
2. Date of Receipt
3. Date of Logging-in
4. Name of Client
5. Number of Samples (not sub-samples)
6. Due Date
7. Completion Date
8. Date of Sample Return to Client
9. Date of Sample Disposal as Waste

The job number consists of a letter followed by a seven digit number. The initial letter code identifies the laboratory (H - Houston, M - Monroeville, C - California). The four digits following the letter identify the year and the month of the sample shipment logging-in, and the last three digits are chronological within the month. Thus, M8709005 is a

job number issued by the Monroeville Laboratory for the fifth job logged in during September 1987.

A sample page of the Master Log is shown in Figure 4-3. At the time of entering the job in the Master Log, the job number is manually written on the Field Chain-of-Custody and on the Sample Shipment Checklist. The job number remains the identification of the job in the laboratory and on the job records.

4.2.5.2 Job Traveller

In addition to opening an entry in the Master Log, a Job Traveller is issued for every job in the laboratory. The Job Traveller is a computer generated document that gives all the pertinent details regarding the individual samples in the job.

For each sample in the job, a unique number is assigned. The unique number consists of the job number followed by three digits, which are sequential within the job. A sub-sample is further identified by a letter following the sample number. Thus, M8709005027C identifies sub-sample C of the 27th sample of job number M8709005.

The sub-sample letter code is used to identify the purpose or the preservative of the sub-sample. The following codes will be used

A	Unpreserved sample for volatiles by GC/MS
B	Preserved sample for volatiles by GC/MS
C	Preserved sample for volatiles by GC/MS
D	Unpreserved sample for volatiles by GC
E	Preserved sample for volatiles by GC
F	Unpreserved sample in glass container for organics
G	Nitric acid preserved sample in plastic for metals
H	Sodium hydroxide preserved sample in glass
I	Acid preserved sample in glass

JOB NUMBER	DATE REC'D	DATE LOGGED	CLIENT	NUMBER OF SAMPLES	DUE DATE	COMPLETION DATE	RETURN (R) DISPOSAL (D)	R/D DATE

FIGURE 4-3

SAMPLE MASTER LOG
PAGE

On the job traveller, each sample is assigned a unique laboratory number. This number is entered on the traveller, as well as the original sample identification off the field chain of custody, the date of sampling, the sample matrix, and the parameters for which the sample or sub-sample are to be analyzed. In addition, the traveller heading contains information pertaining to the job as a whole: the client identification, project contacts, date of logging in, and due date. The traveller also indicates where the sample or sub-sample is stored (refrigerator I.D., shelf number, etc.).

A sample of a Job Traveller is shown in Figure 4-4.

4.2.5.3 Sample and Sub-Sample Numbering

When the computerized logging-in process is complete, the operator entering the information proofs the input and verifies that all the information is correct. The complete sample or sub-sample identifications are entered on labels. The operator then places the correct label on each container of each sample, and verifies once more that the information has been correctly recorded.

The labelled sample containers are then placed in the appropriate storage area as designated on the traveller.

4.2.5.4 Information Distribution and Job Filing

The Job Traveller is the working document for each job. The Sample Management Group personnel makes a copy of each traveller for each section or group in the laboratory, as well as the Data Management Group and the QA/QC Section. The copies are distributed to the group and section managers as required. The original traveller is used to open a file for the job in which the laboratory copy of the field chain-of-custody and the sample shipment checklist are placed. The file is identified by the job number. As the work on the job is completed, the file will be used to store all laboratory records regarding the job.

FIGURE 4-4

(Need a LIMS generated dummy traveller)

The file for the job is placed in the laboratory central active job filing system.

The sample log-in process is extremely critical for the proper functioning of the laboratory. It must be performed rapidly and accurately, so that holding times will not be violated, and so that the correct analyses will be performed on the appropriate samples.

4.2.6 Custody Transfer Within the Laboratory

Because the laboratory is considered a secure facility, samples will be considered as being in the custody of the laboratory from the time that sample receipt is recorded.

All samples will be maintained at the locations designated on the traveller until the section responsible for the sample preparation for analysis is ready to start work on the samples. The section representative will then proceed to recover the samples from their designated storage area. The person retrieving the samples will fill in a transfer form, a sample of which is shown in Figure 4-5, and submit the form to the personnel of the Sample Management Group, while keeping a copy of the form.

When the sample preparation is completed, and residual samples are returned to their original storage location as had been indicated on the Job Traveller, the sample preparation group will return the copy of the transfer form to the Sample Management Group, indicating on the form which samples, if any, have been completely used up. The Sample Management Group will file these forms with the job file.

4.2.7 Sample Disposal

From the laboratory perspective, samples are disposed of when either they are returned to their origination point (the client) or are eliminated as waste.

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KEYSTONE

INTERNAL SAMPLE TRANSFER

Traveller Number	Sample Number	Sample Taken For						
		Organic Extraction	Volatiles Analysis	Metals Digestion	Distillation	Metals Analysis	Anions Analysis	Special (Identify)

Received by:_____

FIGURE 4-5

Date:_____

4-16

Samples that are returned to their point of origination will be accompanied by a chain-of-custody document, and the laboratory will be acting as though it were a field unit. The Sample Management Group will complete a chain-of-custody document as described under field custody in Section 4.1, and ship the samples as described in Section 4.1, unless documented client instructions provide information to the laboratory for a different mode of returning the samples. Copies of these documents will be placed in the job file by the Sample Management Group, and the date of sample disposal will be entered in the Master Log.

Samples that are being disposed of as waste will be recorded by the Sample Management Group on a special form, a sample of which is shown in Figure 4-6. When the disposal is complete, the person disposing the samples will sign and date the form, and enter the date in the Master Log and the form in the job file.

Before samples are disposed of as waste, the Sample Management personnel will review the analytical data and the history of the samples, so that appropriate precautions may be taken. Those samples which were found to be innocuous will be disposed of either as ordinary trash or, if aqueous, by pouring down the drain.

In disposing the samples, as waste, great care must be exercised. Special drums will be maintained for such disposal and the drums will be clearly marked to identify the type of waste that may be placed in each drum. For aqueous wastes two drums will be maintained, the first for acidic waste and the second for alkaline waste. These two types of waste must not be mixed because the acid-base reaction may be too violent, and because the water samples may contain materials that are incompatible with the prevailing pH of the drum (for example, cyanide waste cannot be added to an acid drum because of the generation of hydrocyanic acid).

Two additional drums will be maintained for disposal of organic liquid waste and for disposal of solid samples in which hazardous materials were identified. Samples of solvents, or laboratory waste solvents will be placed in the organic liquid waste drum. Solid samples that have been found to contain hazardous

SAMPLE DISPOSAL RECORD

TRAVELLER NO. _____ DISPOSAL DATE_____

SAMPLE ID	SAMPLE MATRIX	HAZARD					DISPOSAL		
		FLAMMABLE	CORROSIVE ACID	CORROSIVE BASE	REACTIVE	TOXIC	MUNICIPAL SEWER	MUNICIPAL WASTE	DRUM*

*) IDENTIFY DRUM DISPOSED BY _____

FIGURE 4-6

materials, except for strong oxidizing agents, will be placed in the drum for solid waste. Wipes of spills and contaminated sample containers and laboratory ware that are to be disposed, as well as other laboratory waste that may be considered hazardous (such as used syringes) will be placed in the solid waste drum.

For each drum, a log will be maintained identifying in general terms the type and quantity of material that have been placed in the drum. This log will facilitate preparing the manifest for shipping the waste for ultimate disposal.

The drums will be disposed through a contracted firm dealing in the disposal of hazardous waste no less frequently than once per month. While the waste is collected in the laboratory, a special area will be set aside for the storage of the drums.

4.2.8 Custody Transfer for Prepared Samples

When the sample preparation group completes the preparation of a batch of samples, and if the prepared samples (extracts, digests, distillates, etc.) are to be transferred to the analytical group, the sample preparation group completes a transfer form, shown in Figure 4-7. The transfer form is transmitted with the prepared samples to the analytical group, and a copy of the form is given to the Sample Management Group to be placed in the job file.

PREPARED SAMPLE CUSTODY TRANSFER

QC BATCH ID _____ DATE OF TRANSFER _____

EXTRACTS:
B/N/A _____
B/N _____
ACID _____
PEST. _____
O&G _____
TPH _____
OTHER*_____

DIGESTATES:
ICP _____
GFAA _____
CV _____
OTHER*_____

DISTILLATES:
CN _____
PHENOLS ___
F _____
OTHER*_____

TRAVELLER NUMBER	SAMPLE NUMBER	SAMPLE MATRIX	PREP. MATRIX	MATRIX SPIKE

*) IDENTIFY: _____

RELINQUISHED BY _____ RECEIVED BY _____

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FIGURE 4-7

5.0 ANALYTICAL PROCEDURES

In order to produce meaningful results, both sampling and analytical procedures must be sound and complimentary to each other. While close coordination of activities between the laboratory and field services is highly advisable in order to produce a high quality product, the laboratory services will frequently involve analyses of samples collected through organizations other than Keystone. Thus, the application of strict procedures regarding the transfer of samples from the field to the laboratory is not always possible.

5.1 COORDINATION OF ACTIVITIES

Analytical services are requested through several sources. For projects of long duration or of such magnitude as to warrant it, an analytical project manager is assigned. The project manager acts as liaison between the client and the laboratory on all matters pertaining to the project. In other cases, requests for analytical services originate with the laboratory sales representatives, sample control personnel, or other key technical persons in the laboratory. Regardless of the means through which a request has been made, the person receiving the request will enter a description of the requested work on an Analytical Request Form, shown in Figure 5-1, and the information will be entered into the Laboratory Information System as proposed work. When the proposed project becomes a definite task for the laboratory, the Sample Management Group will activate the project and distribute the information together with the anticipated schedule, so that the laboratory sections that will be involved in the analysis are aware of the upcoming work.

All further coordination and scheduling of the work will be issued through the Sample Management Group.

5.2 PREPARATION OF SAMPLE CONTAINERS

For clients and projects that require the laboratory to supply with containers for samples, the Sample Control Group will be responsible for preparing the containers, labelling them, and shipping them to the client. It is the responsibility of the project managers to inform the Sample Control Group of the need for containers. Such

ANALYTICAL REQUEST FORM

Request Taken By: _____ Request Date: _____

Client: _____ Project Manager: _____

Client Contact: _____ Telephone: _____

Address: _____

_____

_____ Start Date: _____

_____

Telephone: _____

NO. OF SAMPLES	MATRIX	ANALYTICAL PARAMETERS	TURNAROUND TIME, DAYS

APPLICATION : [] NPDES [] SDWA [] RCRA [] Superfund [] Other

Special Instructions: _____

_____

_____

_____

_____

_____

FIGURE 5-1

notification must be received by the Sample Control Group at least three working days prior to the time for which the sample containers are required.

For aqueous field samples, new pre-cleaned sample bottles with Teflon lined screw caps will be used. The containers will be prepared with the appropriate preservatives, and will be labelled with information regarding the analytes that are to be determined on the sample.

For solid samples, a variety of containers may be used. For soils, brass sleeves are frequently employed. The brass sleeves will be recycled after thorough cleaning and baking in the laboratory. When the samples are to be collected in polyethylene or glass, appropriate new and pre-cleaned containers will be used.

Table 4-1 lists the appropriate containers and preservatives for aqueous samples.

5.3 INSTRUMENT MAINTENANCE

Maintenance of the instruments in the laboratory in good working order is of paramount importance. Instrument maintenance consists of two major aspects, the first is routine preventive maintenance, and the second is repair due to malfunction.

For all instruments, the manufacturer's recommended schedule of preventive maintenance will be closely adhered to. It is the responsibility of the Section Manager in each section of the laboratory to assure that the time required for preventive maintenance is set aside and planned on. Records of this maintenance will be kept in the instrument log, including information concerning replacement parts, readjustments, and calibrations, which may affect the performance of the instrument relative to its performance just prior to maintenance. Verification of instrument response, and recalibration if necessary, must be performed immediately after the completion of such maintenance, before any sample analysis is taken up.

Instrument repair will be performed as necessary either by a service call or through laboratory personnel. It is the responsibility of the analyst to verify that the instruments are performing within preestablished criteria, and investigate and cause to correct any malfunctions. Instrument malfunction cannot always be detected through routine use; the running of verification samples throughout the course of an analytical

run will be used to further certify that the instrumental response and behavior was within accepted norm.

Should an instrument be serviced, the analyst will verify that the instrument is performing within the required acceptance criteria before analyzing any samples. If the instrument after maintenance is not within these criteria, the instrument will be recalibrated.

Records of all instrument maintenance and repair will be kept in the instrument log. These records will include information regarding the cause of the malfunction, the corrective action that has been taken, record of verification of performance after the malfunction has been corrected, and identification of the samples that may have been affected by the malfunction. The Section Manager, or his designee, will be responsible to assure that the maintenance logs are properly kept and filled, and that notification of invalid data is given to the Data Management Group, if the data from the invalid runs has already been released by the Section. Figure 5-2 shows the form that is used to notify the Data Management Group that certain data are invalid.

5.4 PREPARATION OF STANDARDS

All analytical methods at some point must be validated by the use of calibration standards. A calibration standard is made by the appropriate dilution of a pure substance, the purity of which is traceable to NBS standard. Because of the high sensitivity of many analytical instruments, the calibration standard is an extremely dilute version of the pure compound. Because of the high dilution required, in order to be within the linear range of the instrument, the preparation of the calibration standard is frequently made by serial dilution rather than in a single step. In order to provide standard solutions at sufficiently low concentration, a miniscule amount of the pure substance will be required, the measurement of which is subject to extreme error. Thus, it is preferable to deal with potential dilution errors, rather than with the large error associated with the measurement of a very small amount of the pure substance.

The initial pure standard is usually obtained either as a pure material or already in solution prepared as a certified solution of a given concentration of the pure compound or compounds. In preparing the stock solution of the calibration standard, great care must be exercised in measuring weights and volumes as accurately as possible, since all the

INVALID DATA NOTIFICATION

Laboratory Group:
[] GC
[] GC/MS
[] HPLC
[] Metals
[] Wet Chemistry
[] Miscellaneous

Date of Notification: _____

Parameter: _____

QC Batch No. _____

Data for the following samples, which have been submitted on _____ are not valid.

TRAVELLER NUMBER	SAMPLE IDENTIFICATION

Reason: _____

_____

_____

Submitted by _____

FIGURE 5-2

analyses following the calibration will be based on the accuracy of the calibration, and the accuracy of the ultimate data cannot be any better than that of the calibration curve. Table 5-1 summarizes the valid lifetime of primary and secondary standards used in many tests. These lifetimes should be taken as a guide only. It is the analyst's responsibility to assure that all standards used by him are within the standard solution holding time, and to prepare fresh standard solutions whenever necessary. In preparing working solutions, or using working solutions, the analyst must check for signs of deterioration, such as the formation of cloudiness and precipitation, discoloration. The standard must also be periodically compared with previous runs of standards, and with independently prepared standards to assure that response factors fall within an historically accepted range.

TABLE 5-1

STANDARDS AND SOLUTIONS HOLDING TIMES

MATERIAL	HOLDING TIME		
	PURE COMPOUND	STOCK SOLUTION	WORKING SOLUTION
Volatile organic compounds for GC or GC/MS analysis	1 Yr @ -10°C	2 Mo @ -10°C	1 Wk @ -10°C
Semivolatile organic compounds for GC or GC/MS analysis	1 Yr @ 4°C	1 Yr @ 4°C	6 Mo @ -10°C
Semivolatile organic compounds for HPLC analysis	1 Yr @ 4°C	1 Yr @ 4°C	6 Mo @ 4°C
Pesticides (Cl, P, N) and herbicides	1 Yr @ 4°C	1 Yr @ 4°C	6 Mo @ -10°C
Polychlorinated dioxins and furans	1 Yr @ 4°C	1 Yr @ 4°C	6 Mo @ 4°C
Metals for ICP analysis	Indef. @ RT	1 Yr @ RT	6 Mo @ RT
Metals for GFAA analysis	Indef. @ RT	1 Yr @ RT	6 Mo @ RT
Mercury	Indef. @ RT	6 Mo @ RT	1 D @ RT
Hexavalent chromium	Indef. @ RT	1 Yr @ RT	1 Yr @ RT

For anions and other parameters, the holding times of standard solutions should be checked in the appropriate method.

All standards and standard solutions of organic compounds will be maintained in glass containers, and will be protected from light. The position of the meniscus in each container will be marked after each time that the container is opened, so that changes due to evaporation can be detected.

Metals working solutions and stock solutions will be kept in polyethylene containers at room temperature. The position of the meniscus will be marked each time a solution is used to insure that concentration changes due to evaporation are detected. Before using any standard solution, the analyst will examine it for signs of precipitation and changes in color. If precipitation has occurred, the solution will be discarded and a new standard prepared. Discoloration frequently is only a warning sign, but will not affect the results. If a solution is discolored, the analyst will compare the results with historically established response factors, to assure that the solution is still within the operating range of the method, and within experimental error of its original concentration.

The preparation of standards is very exacting. To facilitate the operation of preparing standards, a separate area is set in the laboratory equipped with a small hood and analytical balance. A freezer in the same room is used to store all primary standards, and no other samples or extracts. In this fashion, the contamination of standards by samples, and vice versa, is minimized.

For each stock standard solution that is prepared, accurate records will be kept in a special logbook used only for the maintenance of standards data. The following information will be entered in the logbook at the time of stock standard preparation:

a. Date of preparation

b. Application for which the standard is being prepared (i.e., identification of the method)

c. For each compound, the supplier of the primary standard, the batch number, and the amount taken

d. The solvent identification (compound, supplier, batch number)

e. The final volume of the stock standard

f. The identification number assigned to the stock standard preparation

g. The name of the analyst preparing the standard

A typical page of the logbook is shown in Figure 5-3.

The identification number that is assigned to the stock standard is alphanumeric consisting of the date preparation and a method code. Thus, 090587MS625 will be the identification number of a standard prepared for semivolatiles by GC/MS on September 5, 1987.

In preparing the diluted working standards, it is the analyst's responsibility to make sure that the stock standard is of valid vintage. The preparation of all working standards is also recorded in the logbook. The following information is recorded in the logbook:

a. Date of preparation
b. Identification number of the stock standard solution
c. Volume of stock standard solution taken
d. Final volume of the diluted standard solution
e. Concentration of each parameter in the diluted standard
f. Identification number for the diluted standard
g. Name of the analyst preparing the diluted standards.

The working standards will also be identified by an alphanumeric system consisting of date, method, and nominal concentration. Thus, 090587MS625C10 would be the identification of a working standard prepared on September 5, 1987, for semivolatiles by GC/MS, and in which the nominal concentration of the components is 10 ng/uL in the working solution. The ppb or ppm designation is intentionally not employed because it usually refers to the concentration of the compounds in water. Since our activities involve identification and quantitation in all matrices, it is preferred to identify the concentration in the calibration solution *per se*.

The working standards for organic analysis will be stored in a freezer in the work area. No other samples or extracts will be stored in the same freezer. The working standards for metals will be stored in the work area in a cabinet or shelf designated for standards only. These need not be refrigerated. For other parameters, the working standards will

SOLUTION ID. NO.	PREP.		STOCK IDENTIFICATION					COMPOUND	QUANTITY TAKEN					FINAL VOL. mL	FINAL CONC. ug/mL
	DATE	BY	NUMBER	PURE COMP.	STOCK SOL'N	CONC. STOCK SOL'N, ug/mL	EXP. DATE		AMOUNT	g	mg	uL	mL		

FIGURE 5-3

STANDARD PREPARATION LOG

be maintained in either a refrigerator or at room temperature in the work area, but at specifically designated cabinets or shelves, where no other materials are being stored.

5.5 DETERMINATION OF DETECTION AND QUANTITATION LIMITS

For many projects, knowledge of detection limits is essential in order to be able to bracket analytical results that are obtained. Several such limits exist, and different experts define these limits differently. For this reason, the definition and determination by which Keystone abides is given below.

5.5.1 Instrument Detection Limit

In simple terms, the instrument detection limit is the smallest quantity of material that the instrument can detect. It has been defined in the past as a certain value of the signal-to-noise ratio. Many modern instruments, however, are designed to self compensate for noise, so that the measurement of the signal-to-noise ratio is not a simple matter.

For the purposes of work at the Keystone Laboratories, the instrument detection limit is defined as three times the standard deviation from the mean of seven replicate measurements of a low concentration standard that produces a definite, measurable signal. The signal may be an area count, a peak height, an absorbance reading, or electric measures (such as voltage, current, resistance). The nature of the signal is dictated by the instrument and detector that are used. The instrument detection limit is calculated from the following equation:

$$IDL = \frac{3S}{RF}$$

Where,

IDL = Instrument detection limit, in weight units (ng, mg) for those parameters where the signal depends on an absolute quantity (such as chromatographic methods), and in concentration units for those parameters that are concentration dependent

S = Standard deviation of the seven replicate readings, in units of the reading (i.e., area count, peak height, etc.)

RF = Response factor, in units of signal reading/unit weight, or concentration, depending upon the units used for IDL.

The calculation of the response factor is shown in Section 5.6, where calibration procedures are discussed. The calculation of the standard deviation is shown below.

$$S = \sqrt{\frac{\sum_{i=1}^{n}(x_i - X_m)^2}{(n - 1)}}$$

where, x_i = The value of the i'th reading of the set of replicates

X_m = The mean value of the replicates

n = The number of replicate measurements

The mean, X_m, is determined as follows:

$$X_m = \sum_{i=1}^{n}\frac{x_i}{n}$$

In order for the results to be useful, the standard chosen to obtain the detection limit should be such that the mean of its readings, X_m, is slightly greater than 3S. This may require some trial and error initially when a new instrument is installed.

The instrument detection limit will be determined on a quarterly basis, and whenever the instrument has undergone extensive maintenance. Records of performing the determination of the instrument detection limit will be kept in the instrument log, and the values of the instrument detection limit will be updated in the working SOP's at each time that the instrument detection limits are determined.

5.5.2 Method Detection Limit

The method detection limit is obtained in a manner very similar to that of the instrument detection limit. The principal difference is that in determining the

method detection limit (MDL), the analytes are subjected to the entire analytical protocol for the specific method that is being employed. Ideally, the method detection limit should be determined for every matrix that is being analyzed. Unfortunately, obtaining reproducible, well characterized matrices for media other than water is not yet feasible. Hence, method detection limits will be determined for water only.

To determine the method detection limit, seven replicates of laboratory pure water are each spiked with a known amount of the analyte. The amount that is being added is the same for all seven replicates, and should be 2 - 3 times greater than the previously determined instrument detection limit. The seven replicates are subjected to the same analytical procedures as a sample would be, and the concentrations of the analytes of interest are measured. The method detection limit, as was the instrument detection limit, is defined as three times the standard deviation of the seven readings. The calculation of the method detection limit should be done in units of weight of the analyte. In this fashion, such variables as injection volume in chromatographic techniques or pathlength in spectrophotometric techniques are eliminated.

The equations that apply to the calculations of method detection limits are identical to those used for the instrument detection limit.

$$MDL = \frac{3s_m}{RF}$$

where

MDL = Method detection limit, in units of weight (ng, ug) for those methods that depend upon an absolute quantity, and in concentration units for those methods that depend upon concentration

s_m = The standard deviation of the seven readings from the mean, in units of signal size (area, height, etc.)

RF = The response factor of the instrument to the analyte, in units of signal size/unit weight or concentration, depending upon the MDL units.

The standard deviation is determined just as before from the equation:

$$s_m = \sqrt{\frac{\sum_{i=1}^{n}(y_i - Y_m)^2}{(n - 1)}}$$

where y_i = the instrumental reading for the i'th sample that has gone through the entire preparation procedure

Y_m = The mean value of the replicate readings

n = The number of replicates that have been run.

The mean value is determined as follows:

$$Y_m = \sum_{i=1}^{n}\frac{y_i}{n}$$

As a minimum, the method detection limit will be determined for all analytes associated with the method on a semiannual basis. It will be determined quarterly for a selected list of analytes, which are normally used in spiking samples. During the quarterly determinations, the method detection limits for analytes that are not specifically analyzed at that time will be determined by applying established ratios to the analytes that are normally used for spiking.

The quarterly determination of method detection limits will also be performed whenever the instrument undergoes major repair or modification, if the measurement of the instrument detection limit shows a significant departure from the previously determined instrument detection limit. If the instrument detection limit has remained substantially unchanged after the repair or the modification, there is no need to run the method detection limit again.

The method detection limits must also be determined whenever the sample preparation mode is modified.

5.5.3 Quantitation Limits

The quantitation limit is determined at the same time as the method detection limit and from the same runs. The quantitation limit (QL) is defined as five

times the standard deviation that has been measured in determining the method detection limit. Thus,

$$QL = \frac{5s_m}{RF}$$

where the symbols have the same meaning as before.

5.5.4 Conversion of Detection Limits to Minimum Detectable Concentration

The conversion of the detection limit (MDL and QL) to a minimum detectable concentration in a sample is done as follows:

$$DLS = \frac{DL}{v_i} \times \frac{v_j}{S}$$

where

- DLS = Detection limit in sample in units of weight per unit weight or per unit volume
- DL = Either the MDL or the QL as defined in the preceding sections
- v_i = Volume of prepared sample taken for analysis (such as the volume of extract injected into a GC), in mL
- v_j = The volume of the prepared sample (such as the final volume of an extract), in mL
- S = The sample size that was taken to produce the prepared sample of volume v_j. Sample size is normally measured in liters for aqueous samples and in grams, dry weight, for solid samples.

5.5.5 Documentation of Detection Limits

Whenever instrument detection limits, method detection limits, and quantitation limits are determined, the results will be copied to the QA/QC Section. The results must identify the type of detection limits, and include both the value in terms of weight (absolute quantity) and concentration. In reporting the concentration units, standard sample sizes and aliquots will be reported.

5.5.6 Application of Limits in Data Reporting

In reporting data, the following rules pertaining to detection limits shall apply:

a. Experimental data which are equal to or greater than the value of DLS that has been calculated on the basis of QL will be reported with the concentrations found, without any qualifiers.

b. Experimental data which are equal to or greater than the value of DLS, calculated on the basis of MDL, but are less than the value of DLS, calculated on the basis of QL, may be reported, with the qualifier that the concentrations are estimated below detection limits.

c. Experimental results below the value of DLS, calculated on the basis of the MDL, will be reported as not detected at the DLS value.

Exceptions to these rules will be applied only for contracts and projects which specify their detection limits, and whose detection limits exceed the value of QL. Also exceptions will be applied when a prepared sample requires very high dilution in order to have the analyte of the highest concentration within the linear range of the method. In the latter case, the client will be so notified, so that decisions can be reached if the client needs additional dilutions measured.

5.6 INSTRUMENT AND EQUIPMENT CALIBRATION

Instrument and equipment calibration must be rigorously and routinely performed in order to provide reasonable assurance that the data generated are valid and acceptable.

Two principal types of calibration are performed. The first is an initial calibration, which consist of determining the linear range of the instrument and its response factor. The second is a verification calibration, which serves, during the course of running samples, to ascertain that the instrument calibration has not drifted unacceptably. The frequencies of performing the different types of calibrations are shown in Table 5-2.

5.6.1 Initial Calibration

All instrumental methods of analysis are subjected to an initial calibration, consisting of the measurements of responses to five different standard solutions

of the analytes of interest. The standard solution of the lowest concentration should have a concentration of the analytes of interest approximately 2 - 3 times the concentration that corresponds to the Instrument Detection Limit; and the standard solution of the highest concentration should have a concentration of the analytes of interest at or near the upper end of the linear range of the method.

In performing the analyses of the standards to determine the response factor and the linear range, the standard solution is prepared as mentioned in Section 5.4, and surrogates and internal standards are added to it when appropriate. The identification of the working standard solution and the date of performing the run are entered on the records of the run by the analyst.

When the five runs are completed, the responses are fitted to a straight line of the form

$$y = ax + b$$

Where y = the measured response
x = the known amount of the analyte
a = the response factor
b = the y-intercept.

In addition to determining the values of a and b, the correlation coefficient is determined. The latter is a measure of how closely the five points were to the straight line. The correlation coefficient is determined from the following equation:

$$r = \frac{n\sum_{i=1}^{n} x_i y_i - \sum_{i=1}^{n} x_i \sum_{i=1}^{n} y_i}{\sqrt{n\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i\right)^2}\sqrt{n\sum_{i=1}^{n} y_i^2 - \left(\sum_{i=1}^{n} y_i\right)^2}}$$

where r = Correlation coefficient
x_i = The known amount of the analyte in the i'th standard run
y_i = The measured response to the to the i'th standard
n = The number of standards run to obtain the calibration curve.

In order for the calibration curve to be valid, the correlation coefficient must be 0.995 or higher, and the ratio b/a must be no greater than the method detection limit (MDL). If the correlation coefficient is not met, it usually implies that either the lowest or the highest concentration of standard is outside the linear range. To correct for this, the analyst should rerun the highest standard, and also run a high standard somewhat more dilute than the initially used highest concentration. Similarly, the analyst should examine the effect of increasing slightly the concentration of the lowest standard.

If the ratio b/a criterion is not met, the problem may be with contamination in the system or change in the noise level of the instrument. To correct for this, the instrument detection limit should be first checked. If it has in fact changed, the ratio should be compared to the newly determined noise level, in order to see if the criterion is met.

While, as much as possible, certifiable standards are used in the preparation of solutions for calibration, it is always possible that the manufacturer has made a mistake. To circumvent the possibility of erring due to a mistake in the manufactured primary standard, a check sample will be analyzed whenever an initial calibration curve is constructed. The check sample will consist of a solution of the analytes of interest, and at known concentration, but obtained and prepared by a different source than the manufacturer of the calibration standards. When the analyte concentrations in the check sample are calculated, they should differ by no more than 20% from the known concentration. If the discrepancy is greater than 20%, a determination of the source of inaccuracy will be performed.

Once the initial calibration curve has been determined and verified, a table is prepared with the response factors for all the analytes. The table also includes the identifications of all the standards used in generating the data, and the date of running the initial calibration. A copy of the table is submitted by the analyst to the QA/QC Section, and another copy is maintained in the work area for ready reference on a daily basis.

5.6.2 Continuing Calibration

Continuing calibrations, sometimes also called verification calibrations, serve to insure that the instrument, during the course of running samples, is remaining sufficiently stable so that the response factor calculated in the initial calibration remains valid.

In performing a continuing calibration, the analyst analyzes a midrange standard, containing all the analytes of interest and internal standards and surrogate compounds if applicable. The response factor is determined for each analyte by dividing the signal by the known concentration of the analyte. If the response factor is within 10% of the originally determined response factor, the instrument is considered to be within calibration, and analysis may continue without performing the initial calibration procedure again. If the response factor is determined to be outside the acceptance range, the instrument will be recalibrated by using the initial calibration process. Samples that have been analyzed since the last acceptable calibration will also require to be reanalyzed, after the instrument has been recalibrated.

In recording the information on continuing calibrations, the analyst will enter the identification of the initial calibration to which the continuing calibration refers back, and a list of the samples that have been analyzed since the previous acceptable continuing calibration. A copy of this information will be submitted to the QA/QC Section so that the data associated with the calibration can be validated. Another copy or copies will be submitted to the Data Management Section for inclusion in the file of the project from which samples have been analyzed while the specific continuing calibration was in force.

At no time should the response factor be corrected on the basis of the continuing calibration. Until such time as it is necessary to reestablish the initial calibration, the response factors determined in the initial calibration will be adhered to.

5.6.3 Calibration Frequency

Instruments have widely variable stabilities, requiring variable frequencies of calibration. Table 5-2 summarizes the frequencies of such calibrations. It should be emphasized that these frequencies are based on instruments that are performing normally. It is or should be obvious that after change in instrument parameters or after repair, the initial calibration must be repeated, and the cycle started over again. Thus, the frequencies included in the table are minimum requirements.

TABLE 5-2
CALIBRATIONS FREQUENCIES

INSTRUMENT	APPLICATION	INITIAL CALIBRATION	CONTINUING CALIBRATION
GC/MS	Volatiles	Once per Week	Every twelve hours and at the end of a sequence of runs if the instrument is about to be idle.
GC/MS	Semi-Volatiles	Once per Week	Every twelve hours and at the end of a sequence of runs if the instrument is about to be idle.
GC	Volatiles by Purge-and-Trap	Every Three Days	After every eight runs, and at the end of a sequence of runs.
GC	Extracts	Every Three Days	After every eight runs, and at the end of a sequence of runs.
HPLC	Extracts	Every three Days	After every eight runs, and at the end of a sequence of runs.
IC	Solutions	Every Three Days	After every eight runs, and at the end of a sequence of runs
Autoanalyzer	Solutions	Daily	After every ten runs, and at the end of a sequence of runs
ICP	Digests	Daily	After every ten runs, and at the end of a sequence of runs
AA	Digests	Daily	After every ten runs, and at the end of a sequence of runs.
Mercury Analyzer	Digests	Daily	After every ten runs, and at the end of a sequence of runs.

TABLE 5-2

CALIBRATIONS FREQUENCIES
(CONTINUED)

INSTRUMENT	APPLICATION	INITIAL CALIBRATION	CONTINUING CALIBRATION
TOC Analyzer	Solutions	Daily	After every eight runs, a[illegible] the end of a sequence of [illegible]
TOX Analyzer	Solutions	Daily	After every eight runs, a[illegible] the end of a sequence of [illegible]
IR	Solutions for O&G or TPH	Daily	Every ten runs, and at the end of a sequence of runs.
UV/Vis Spectrophotometer	Solutions	Daily	Every ten runs, and at the end of a sequence of runs.
Fluorometer	Solutions	Daily	Every ten runs, and at the end of a sequence of runs.
pH Meter	Aqueous Solutions	Daily[1]	Every ten runs, and at the end of a sequence of runs.
Analytical Balance	Solids	Annual[2]	Daily[2]
Thermometers	Temperature Measurement	Quarterly[3]	Not applicable

1.) pH calibration requires the use of only three standards.

2) Analytical balances are calibrated and serviced annually by representatives of the manufacturer.Their calibration is checked daily before any weighings are done against a certified class S weight. Deviations from the true weight are recorded and applied to weighings when appropriate.

3) Thermometers are calibrated quarterly against an NBS certified thermometer, and all required corrections are applied to measurements.

5.7 ANALYTICAL METHODS

Whenever possible, the analytical methods that will be employed are either EPA, NIOSH, AOAC, or ASTM. The abbreviated methods are included in the Standard Operating Procedures, and complete copies of the methods are maintained in each laboratory. They will not be reproduced here.

5.8 ANALYSIS OF QUALITY CONTROL SAMPLES

Routine quality control samples are analyzed to assure that the operation is within control as established for the laboratory on the basis of historical data. The routine quality control consists of blanks, spiked blanks, spiked sample, duplicate sample, and external check sample analyses. These are discussed separately in the following sections.

5.8.1 Blanks

Four types of blanks may be associated with any batch of samples. These are: reagent blank, method blank, trip blank, and field blank. The latter two types are considered by the laboratory as ordinary samples, and are not therefore part of the internal laboratory QC, although they are very much part of the program QC. Thus, trip and field blanks will not be discussed here.

5.8.1.1 Reagent Blank

Reagent blanks are set aside whenever the reagents are used for preparation of samples. The reagent blank is the reagent or group of reagents that is normally used for sample preparation, without going through any of the preparation steps. This reagent blank is normally not analyzed, unless the method blank, discussed in Section 5.8.1.2, shows the presence of contamination which may have arisen from the reagents. The reagent blank will be labelled with the QC batch identification, followed by the letter R. It will be set aside until all the samples of the QC batch have been analyzed. At that point, if the method blank was acceptable, the reagent blank may be discarded.

5.8.1.2 Method Blank

The method blank is a preparation carried through the entire preparatory steps, except that the reagents do not come in contact with a sample. Rather, laboratory reagent water is used in lieu of a real sample. Ideally, the method blank would be prepared using a matrix that is similar to the

matrix of the samples that are being prepared. Since a reference matrix is not available for matrices other than water, water is the only matrix used as a method blank.

In preparing the method blank, water is spiked with surrogates and internal standards if appropriate for the method, and the water sample is carried through the entire analytical procedure. The method blank is prepared with every batch of samples that is being prepared at the same time, provided the batch is no greater than twenty samples. For batches which are greater than twenty samples, a method blank will be prepared for every sub-batch of twenty samples. In addition, a method blank is prepared whenever the lot number of any of the reagents is changed. The preparation log will then indicate which samples are associated with the new lot number of reagents.

The method blank is analyzed before any samples are analyzed, and the data of the analysis are reviewed. If no analytes are found above the method detection limit, analyses of the prepared samples may be undertaken. If analytes are found above the method detection limit, but below the quantitation limit, the associated prepared samples may be analyzed, but the results will be corrected by the blank.

If analytes are found above the quantitation limit, analyses of the associated samples will not be undertaken until the contamination source is identified and isolated. At this stage the reagent blank will be analyzed. If it is found that the reagent blank shows the contamination, the samples will be reprepared using a new lot of reagents.

5.8.2 Spiked Blank

The spiked blank, or laboratory control standard, serves as a measure of accuracy of the analytical procedure in the laboratory. The spiked blank is prepared by adding prescribed amounts of specific analytes to laboratory reagent water, prior to preparation of the water for analysis. For inorganic parameters,

a spiked blank is prepared for each batch of twenty or fewer samples that are prepared at the same time. Water is spiked for every analyte of interest, for the inorganic parameters.

For organic parameters, a spiked blank is prepared for every batch of samples that is subjected to sample preparation at the same time. The spike contains only selected analytes, which are specified in the pertinent methods of analysis in the Standard Operating Procedures.

Preparation of the spiking mixture is done in the same manner as the preparation of standard solutions for calibration. The spiking mixture is then assigned an identifying code, which is recorded at the time of preparing the spiked blank.

The spiked blank is carried through the entire analytical procedure, and the concentrations of the spiked analytes determined. These values are accumulated by the QA/QC Section to update acceptance criteria and to validate a set of runs. The spiked blank forms the backbone of the determination of the reproducibility of data in the laboratory, since it is based on a well-characterized matrix (water) and is designed to be essentially free of matrix effects. If the spiked blank does not meet the established acceptance criteria, it is assumed that sample preparation or analysis have been faulty, and the batch of samples associated with the spiked blank will be reprepared and reanalyzed.

5.8.3 Spiked Sample

Spiked samples serve to identify if the sample matrix provides certain effects which preclude the ability to recover analytes through the prescribed method. Thus, the spiked sample is used only to determine matrix effects.

One sample per batch of twenty or fewer samples, of the same apparent matrix, will be spiked with a spiking solution, in the same manner as the spiked blank. The spiked sample will be processed through the analytical scheme, and the recovery of the spiked analytes will be determined.

In utilizing the information, great care should be taken because deviations from acceptability may be due to procedure or to matrix effects. Generally, if the spiked blank associated with the batch exhibits acceptable recoveries, it is assumed that the sample preparation and analysis have been performed correctly. It is possible, however, that for some reason the specific spiked sample has been handled differently. Hence, a repeat preparation will be used to demonstrate if the analysis or preparation has been faulty. If the repeat analysis shows the same anomaly, it will be assumed that some matrix effects exist which prevent the sample from behaving in the expected manner.

Since sample spiking is performed before the sample is initially analyzed, it is possible that for some parameters the innate level in the sample is so high that the spiked amount is insignificant. Under those circumstances, the analyte spike recovery will not be calculated, but analysis of a spiked sample will not be repeated.

In selecting a sample for spiking, every effort will be made to choose a field sample, and not one of the field or trip blanks. This can be accomplished only if the identity of the blanks is known in advance. If the samples are submitted entirely as blind samples, then the selection of the sample to be spiked will be random.

5.8.4 Sample Duplicate

Sample duplicates are run to assess precision of the laboratory work. One sample in a batch of twenty or fewer samples, of the same apparent matrix, is prepared and analyzed in duplicate.

The purpose of this analysis is to obtain data on the precision of the analytical procedures that are being followed. Clearly, duplicate analyses will provide precision data only for analytes detected in the samples. Thus, development of historical background for some less commonly found analytes may require considerable time.

As in the case of the spiked sample analysis, the choice of sample for duplicate analysis will be limited to actual field samples, excluding blanks, unless the identity of the blanks is not known, in which case the selection will be random.

If the results of the duplicate analyses are not within accepted criteria, the analysis of the batch of samples with which the duplicate samples have been associated will be repeated.

5.8.5 External Quality Control Audit

An external quality control audit will be performed periodically by submitting for analysis known standard materials from a source other than that from which the calibration standards are prepared. These quality control audit samples will be submitted for analysis by the QA/QC Section, and they will be analyzed by the same procedures as are used for the analyses of samples.

The external quality control audit will be performed on a quarterly basis. If there are projects in the laboratory which have these audit samples submitted by the project, and where the results are made known to the laboratory, then the QA/QC Section will not submit the external quality control audit samples for those parameters which are included in the project audit samples.

5.8.6 Record Keeping on Analysis of QC Samples

The results of the analyses of the QC samples may pertain to more than one project, since the sample preparation area batches samples from potentially several sources to provide for efficient operation. Hence, the retrieval of the QC data may be necessary for several different projects. The QC data, however, is identified by the batching identification provided upon the preparation of the samples. Hence, the retrieval of the data can be readily achieved by identification of the preparation batch number.

For every batch of samples, the identification of the batch will be entered at the preparation stage on the appropriate forms. Sufficient number of copies of the forms will be made to file with every project associated with the batch. The

results of the analysis of the QC samples will be filed sequentially by the QC batch number and will be retrieved through the batch number system whenever it is necessary to retrieve these data.

On a routine basis, the QC approval of an analyzed batch will be based on the acceptance of the QC samples associated with the batch. Once determined by the QC Department to be within acceptable limits, the entire batch of samples will be released and the analytical data for each sample recorded with the appropriate project.

5.9 USE OF SURROGATES

The use of surrogates in organic analysis serves as an additional measure of the acceptability of the results. The significant advantage of the use of surrogates is in measuring recovery against an historically established acceptance range in the performance of each analysis. Thus, the data do not depend solely on the spiked blank to assess the quality of each run.

Surrogates are compounds that are expected to behave analytically in a manner similar to the target analytes. The surrogates are added into the sample before the preparation stage is initiated, and their recovery is a measure of the efficiency of the extraction. The following surrogates have been used at the Monroeville Laboratory and are recommended for use throughout the laboratory system.

TABLE 5-3
SURROGATE COMPOUNDS AND ACCEPTABLE RECOVERIES

EPA METHOD	COMPOUND	RECOVERY RANGE
501/601/8010	Bromochloromethane	70 - 120
	2-Bromo-1-chloropropane	70 - 120
	1,4-Dichlorobutane	70 - 120
502/602/8020	Benzotrifluoride	80 - 120
604/8040	2-Fluorophenol	21 - 100
	2,4,6-Tribromophenol	10 - 123

TABLE 5-3

(Continued)

SURROGATE COMPOUNDS AND ACCEPTABLE RECOVERIES

EPA METHOD	COMPOUND	RECOVERY RANGE
605/8050	1-Naphthylamine	10 - 94
606/8060	Dibutylchlorendate	24 - 154
608/8080	Dibutylchlorendate	24 - 154
	2,4,5,6-Tetrachloro-m-xylene	25 - 125
609/8090	2-Fluorobiphenyl	43 - 116
610/8310	Benzo(e)pyrene	43 - 116
	Decafluorobiphenyl	40 - 140
611/8110	Isodrin	33 - 141
612/8120	Dibutylchlorendate	24 - 154
615/8150	2,4-DB	40 - 140

For GC/MS, standard EPA surrogate compounds will be used for all analyses.

5.10 ESTABLISHMENT OF ACCEPTANCE CRITERIA

Establishment of acceptance criteria is necessary in order to be able to determine regularly whether or not quantitative data generated by the laboratory are within the control limits. The principal criteria that are used to measure the quality of the data are accuracy and precision.

The initial determination of acceptance criteria hinges upon repetitive measurements of prepared spiked solutions, and determination of spike recovery. Twenty samples of laboratory reagent water are spiked with the analytes of interest at a concentration of approximately twice the quantitation limit. Where applicable, the water is also spiked with surrogates and internal standards. The samples are then prepared for analysis following precisely the appropriate protocol, and the concentrations of the analytes are determined. From these values, the mean and the standard deviation for the recovery of each analyte are determined. The deviation of the mean from the known spiked amount is

a measure of the accuracy of the method. The standard deviation of the series of measurements is a measure of the precision of the method. The percent recovery is calculated as follows:

$$R = 100 \times \frac{C_m - C_1}{C_s}$$

where R = Percent recovery of the analyte

C_m = The measured concentration of the analyte

C_1 = The native concentration of the analyte in the sample ($C_1 = 0$ for blank spike)

C_s = The amount of analyte spiked into the sample.

The accepted recoveries of the analytes must be within three standard deviations of the mean. It should be emphasized that recoveries are dependent upon both the method of sample preparation and the sample matrix. Thus, recoveries from soil are not expected to be within the acceptance limits as determined for water. Similarly, extraction by sonication may not show the same recovery as would an extraction via a Soxhlet extractor. Thus, acceptance criteria must be determined matrix by matrix, and method by method.

Frequently, samples are spiked at the time of sample preparation, without knowing if the analytes that are being spiked into the sample are present or not, and without knowing if these analytes are at levels that would make the spike amount insignificant. The analyst is cautioned that recoveries of spikes should not be calculated if the amount in the sample is overwhelming the spike. For most applications, if the ratio C_s/C_1 is not equal to or greater than 5, the spike recovery should not be calculated, since the uncertainty in the native concentration is sufficient to cause greater uncertainties in the spike recovery.

The acceptable precision range is defined in a similar manner. The precision is a measure of the deviations from the mean of repetitive measurements. Thus, standard deviation will be used as a measure of precision. More frequently, the relative percent deviation will be used because at best, measurements are performed in duplicate. The relative percent deviation is determined by the equation

$$\%RPD = 100 \ X \frac{x_1 - x_2}{x_m}$$

where x_1 = high value for the analyte

x_2 = low value for the analyte

x_m = mean value for the analyte $= \frac{x_1 + x_2}{2}$

The results of the determination of recoveries and relative percent deviations will be plotted, indicating the upper and lower limits of acceptance, and the upper and lower warning limits. Future data will be considered acceptable if recoveries of spikes and relative deviations of duplicates fall within the acceptance criteria. The control charts will be updated regularly, generally requiring 20 data points to obtain a valid control chart. The QC Section will maintain the control charts and update them regularly, when sufficient data are collected. Whenever the control charts are updated and the acceptance limits modified, the QC Section will issue the new limits to the analytical section of the laboratory in which the analyses are performed. It is the responsibility of the section managers to assure that data within their section are within the acceptance limits. If they are not, corrective action will be initiated immediately by the section manager of the appropriate section.

If five successive measurements, while falling within the control limits, appear on the same side of the mean, the analyst will stop to investigate if the trend indicates that a change in methodology has occurred. Such successive points may indicate a pattern, and it would be necessary to institute a return to preexisting conditions to avoid the possibility that out of control situations may arise.

It is not feasible, in organic analysis, to obtain control charts for every analyte. Thus, while initial control charts are constructed for all the parameters that are being analyzed, continuous verification of the control is obtained through the use of surrogate compounds and the spiked blank analysis. All other analytes in the organic analysis will be assumed to be within control if their relative response to the internal standards and surrogates have remained constant.

While accuracy and precision form the backbone of the acceptability of quantitative data, qualitative identification is more difficult to cast into quantitative measures. In organic

analysis, the principal criterion for chromatographic analysis is the retention time, or relative retention time. Relative retention time is used with those methods employing internal standards. It is the more reliable measure because it is less dependent on such physical parameters as the length of the column. In all cases, the retention time for each analyte, or the relative retention time for the analyte, will be based on the data obtained from the nearest standard.

To determine the acceptance windows for retention times, the continuing calibration data will be employed. For each compound, the retention times obtained in performing the continuing calibrations during the period of one week will be averaged and their standard deviation determined. The acceptance window will consist of three standard deviations from the mean retention time for each compound. The retention time acceptance window will be redetermined whenever the chromatographic column is changed or the chromatographic conditions altered. It is the responsibility of the analyst to maintain the records for retention time criteria. Copies of the established acceptance windows will be submitted by the analyst to the QC Section for reference in reviewing work.
In mass spectrometric analysis, in addition to the retention time, the mass spectral match of the compound to the standard will be used to verify its identity.

In inorganic analysis, the determination of a positive signal, after correcting for known potential interferences will constitute a positive identification.

5.11 DEVELOPMENT OF NEW OR MODIFIED METHODS

There are occasions when it is necessary in the laboratory either to devise a new approach to analysis or modify an existing method. The former may be needed for analytes for which proven methods do not exist, or if they do, they are not applicable to the matrix being handled. The latter case is frequently the situation that arises because of unusual matrix interferences. It is not the intent of this manual to prescribe analytical approaches to analytes for which methods do not yet exist. However, in order that the laboratory may use the methods with any reliance on the data, the new method must be subjected to the repetitive analyses of a spiked matrix in order to ascertain the precision and accuracy of the method. No method will be employed by the laboratory without the establishment of precision, accuracy, and detection limits.

When a method is being modified, or a new method is being devised and tested, for analytes for which an existing approved method is available, the new or modified method will be subjected to equivalency testing. In performing the equivalency testing, the analytes will be subjected to seven replicate analyses in parallel by the existing method and the new method. To be acceptable, the new method must produce results that are statistically equivalent, or are better than the old method. In addition, sufficient replicate analyses will be performed to assure that acceptance criteria may be established. The new or modified method will then be written, the method with the supporting data and documentation will be reviewed by the Section Manager for the section in which the analyses will be performed and by the QA/QC Manager. After correction and amendments are inserted, the QA/QC Section Manager will transmit the information and the new method to the Technical Director for approval. When approved by the Technical Director, the method may become part of the repertoire of the laboratory.

6.0 DATA HANDLING

The data produced by the various sections of the laboratory are ultimately the product which the laboratory offers. Hence, not only is it necessary to produce results with accuracy and precision, but it is also necessary to be able to maintain the traceability of the data and the association of sets of data with each other. The responsibilities to the production of accurate and precise data are with the individual analysts. They are, after all, the producers of the data. No amount of supervision and validation can correct for mistakes or omissions occurring at the bench. At best, supervision and validation can cull the unsupported data. Traceability of the data, however, pertains to the manner in which the records are kept within the laboratory as a whole. Because of this, record keeping in the laboratory, will be addressed first. This will be followed by a brief discussion of data reduction, data validation, data review and release, and finally reporting.

6.1 DATA RECORDING

The modes of maintaining data in the various sections differ with the methods of analysis. Certain aspects of record keeping have been addressed in the section pertaining to the sample logging and distribution of the information through the laboratory. The traveller and the sample transfer forms (whether for prepared samples or for raw samples) constitute the first step in the generation of data for any set of samples. Samples in the laboratory, however, are analyzed in batches. On large jobs, a batch may constitute just a portion of the total number of samples in the job. For small job, a batch may consist of samples from several travellers handled together. Thus, the maintenance of records in the laboratory must also provide for cross-referencing batches and travellers.

6.1.1 Notebooks

Certain records are maintained in notebooks by the analysts. These records may pertain to methods that are entirely manual, such as many of the methods in wet chemical testing, or they may be used to record unusual observations during the performance of preparation and analyses of instrumented techniques. These notebooks become a permanent record of laboratory work, and they must be traceable.

Bound hardcover notebooks, with prenumbered pages, will be numbered and issued by the QA/QC section. The QA/QC section will number the notebook, and record in its own notebook log the date of issuance and the laboratory section to which the notebook has been issued. When the notebook has been filled, it will be returned to the QA/QC section for permanent archiving.

Users of the notebooks will maintain good laboratory practices in their use. No pages will be torn out of the notebook; corrections will be done by single line-out erasures, followed by initialling and dating the correction; and each page of the notebook will be signed and dated by the analyst at the time of filling the page. The use of such materials as tape or liquid paper to make corrections is not permitted.

6.1.2 Record Keeping in Sample Preparation

Sample preparation for analysis is generally a manual effort. The records in this operation are manually kept. All records for routine sample preparation will be kept on standardized forms. The forms will be filled by the person preparing the samples. The forms will be filled in ink, with no erasures.

If an error occurs, the preparer will line out the erroneous entry once, and enter the correct entry. The preparer will then initial and date the correction.

The following information must be entered:

a. The analysis type for which the samples are prepared (usually the heading of the sheet)

b. The date of preparation, and the name of the preparer

c. The QC batch identification

d. A listing of the samples being prepared, using the Keystone sample identification

e. For each sample listed, the quantity of sample taken, including the units of measurement (for solid samples, use the wet weight)

f. If the method calls for the addition of surrogates, then for each sample the

surrogate mixture identification and the quantity, including units, will be entered

g. For any sample that is spiked, the identification of the spiking mixture and the quantity, including units, will be entered

h. The identification of the medium of the prepared sample (i.e., the solvent for organic extraction, the acid for metal preparation, etc.) and the lot numbers of the reagent media

i. The final volume of each prepared sample, including units

j. Any unusual observations. If these are recorded in a notebook, the notebook and page numbers must be entered on the form.

In addition to filling the form, the preparer must also tag the prepared samples. As a minimum, the tag will identify the sample and the QC batch in which the sample has been prepared.

When the preparation is complete, the form will be checked for completeness by the supervisor of the section, or his designee, and initialed and dated. The preparer will make as many copies of the filled form as there are different jobs in the batch. The original of the form will be maintained in the file of the preparing section. The preparer will then transfer the prepared samples with the accompanying copies of the preparation records to the section responsible for the analysis of the prepared samples.

Standard forms for maintaining the records in the sample preparation areas are shown in Figures 6-1 and 6-2.

6.1.3 Record Keeping in Instrumented Analysis

Many instrumental methods of analysis produce printed traces, charts, or tables. Some of the information specified below is entered through a data system before the runs are made. Clearly, those items of information need not be reentered manually.

For every technique, however, a run log must be maintained. The following information must be entered in the run log by the analyst:

ORGANIC EXTRACTION

DATE ____________________ BY ____________________

QC BATCH NO. ____________________

MATRIX ____________________

METHOD (MARK ONE)

- [] EPA 3510 Sep. Funnel
- [] EPA 3520 Continuous
- [] EPA 3540 Soxhlet
- [] EPA 3550 Sonication
- [] Other, Specify ____________

COMMENTS ____________________

Reagents, Solvents, and Clean-Up Materials:

Material	Mfr	Lot No.	Activation

SAMPLE ID	AMOUNT (circle: mL, g)	SURROGATES		SPIKING MIX		FINAL SOL'N		CLEAN UP	COMMENT note-page
		Mix ID	Vol., uL	Mix ID	Vol., uL	Solvent	Vol., ml		

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FIGURE 6-1

METALS PREPARATION

DATE ______________________

QC BATCH NO. ______________

MATRIX ____________________

BY ________________________

METHOD (MARK ONE)
- [] EPA 3005 Aq. for Flame and ICP
- [] EPA 3010 Aq. for Flame and ICP
- [] EPA 3020 Aq. for GFAA
- [] EPA 3040 Oil, Grease, Wax
- [] EPA 3050 Sed., Soil, Sludge
- [] EPA 7470 Aq. mercury
- [] EPA 7471 Mercury in solids
- [] Other, Specify ______________

COMMENTS ______________________

Reagents:

Material	Mfr	Lot No.

SAMPLE ID	AMOUNT (Circle: mL, g)	SPIKING MIX		FINAL SOLUTION		COMMENTS note-page
		Mix ID	Vol., mL	Matrix	Vol., mL	

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FIGURE 6-2

a. Identification of the analysis (usually this will be the heading of the log)

b. Identification of the instrument

c. Date of analysis, and identification of the analyst

d. Identification of the QC batch

e. Sequential listing of runs (including tuning, initial calibration or continuing calibration, blanks, spiked blanks, samples, duplicate samples, spiked samples, etc.). This listing must be accurate and sequential, and the run number (where applicable) must be included

f. Aliquot of prepared sample that is taken for analysis, including units

g. Any unusual observations. If these are entered in a notebook, they need only be summarized on the form, and the notebook and page numbers entered for further identification of the comments.

Where printouts for each single run are obtained separately, the printout must include the identification of the method of analysis, date of analysis, identification of the analyst, identification of the run, identification of the QC batch, and the aliquot of sample that was taken.

Before transferring the data further, the analyst must confirm that the results as they appear on the printout are correct. If the results are incorrect, the analyst will line them out once and enter the correct value manually. These corrections will be done in ink. When the analyst is finished with the batch of samples, including the review of the data, he will initial the run log and transfer the run log with the data to the Section Supervisor.

The section supervisor will review the run log to verify that the sequence of runs has been properly followed, as required by the specific method. The section supervisor will also review the QC data pertaining to the batch, and verify that the instrument was performing within the required specifications. If any runs appear to have caused problems, the section supervisor will make a point of checking those data; otherwise, the section supervisor will only spot check the results on about 20% of the sample runs.

When the section supervisor is satisfied that the data are valid, he will initial the run log and date it. He will have as many copies made of the run log as there are

jobs represented in the batch that has been analyzed, plus one additional copy for the QA/QC section. He will then transfer the samples data to the data management group, and the QC data to the QA/QC section. Together with the data, the section supervisor will transfer the copies of the records from the preparation section to the data management section and to the QA/QC section.

The original run log will be maintained in the files and records of the section responsible for the analysis.

For instrumental methods which do not produce the identifying code of the run on the permanent records, such as methods that employ a strip chart as the instrumental output, the individual traces will be identified manually by the run number, and the identification of the parameters associated with the run will be recorded manually on the run log.

6.1.4 Record Keeping in Noninstrumented Analyses

Manual analyses have become a rarity in the modern laboratory. Nonetheless, such techniques are still occasionally employed and the data generated by these methods are useable. Raw data from these analyses will be kept in hardbound notebooks, including all the pertinent information that is normally recorded on printouts in other technologies. The results will be summarized on a summary form and submitted to data management and to QA/QC section. The summary form will include an identification of the notebook number and page numbers within the notebook that pertain to the analysis.

6.2 DATA REDUCTION

Reducing the raw data to a presentable form is the responsibility of the analyst performing the analysis. In reducing data, the analyst must take into account whether the sample is aqueous or solid, the sample size, and whether or not the data are to be presented in the form of wet weight or dry weight. All final values must be accompanied with the units of the value. The following equation applies to the calculation for concentration of an analyte in most analytical techniques employed in the laboratory.

$$C = \frac{I}{RF} \times \frac{1}{v_i} \times \frac{v_e}{A_s} \times DF$$

where, C = Concentration of the analyte in the sample, in appropriate units [ng/L (ppt), ug/L (ppb), mg/L (ppm); ng/kg (ppt), ug/kg (ppb), mg/kg (ppm)]

I = Signal size, in units appropriate to the method

RF = The response factor, as defined in Section 5.6.1, in units of signal size per unit weight of the analyte. This response factor is essentially a mean response factor, determined through regression of the initial calibration data. If it is not possible to use the mean response factor (usually because of instrumental software limitations), then the response factor from the run that corresponds to the midpoint of the linear range is used, as long as this response factor does not differ from the mean by more than 10%. The response factor to be used is always taken from the initial calibration.

v_i = The aliquot size (such as injection amount) of the prepared sample, taken for analysis, in units of mL.

v_e = The total final volume of the prepared sample (extract volume, digest volume, etc.) in units of mL.

A_s = The amount of sample taken for preparation. For liquid samples, use mL; for solid samples use the weight in kg. If the results are to be determined on the basis of dry weight, use the following to determine the sample size:

$$A_s \text{ (dry)} = A_s \text{ (wet)} \times \frac{\%\text{solids}}{100}$$

DF = Dilution factor. The dilution factor is 1 for samples that are prepared exactly as prescribed in the protocol. If the extract or digestate require dilution, then the dilution factor differ from unity. For example if an extract is diluted from 1 mL to 100 mL, the dilution factor becomes 100. If an extract is concentrated from 10 mL to 1 mL, the dilution factor becomes 0.1.

In many methods, the data reduction is computerized, alleviating the need for extensive manual reduction of the results. Computers, however, are not perfect. The analyst must review the data, relating it back to the fundamental measurements on which the analyses are based. Thus, in chromatographic techniques, the analyst will compare area counts of peaks with those of the corresponding standard, and verify that the data were in fact correctly reduced. It is also the respnsibility of the analyst to verify the identification of parameters.

It is of the utmost importance that the analyst pay close attention to the data being reduced by him. Data are only spot checked beyond the analyst.

6.3 DATA VALIDATION

Data validation within each analytical section has been discussed previously. Before data from an analytical batch may be incorporated into reports, the validation process outside the laboratory section that produced the data consists of a review of the QC data on the batch by the QA/QC section. It is not the responsibility of the QA/QC personnel to check and verify every value generated and reported by the laboratory.

In reviewing completed batches, the QA/QC personnel will check for the following items, using a checklist to record their observations:

a. Is the batch complete

b. Have all the analyses been performed within holding times, and if not is there an acceptable explanation for deviations from the holding times

c. Is there a valid continuing calibration associated with the runs for each parameter of the individual samples within the batch

d. Is the sequence of runs in which the samples were analyzed proper for the method (i.e., are there regular blanks run when necessary, are there standards run for methods requiring periodic standards, are there laboratory blank spikes and matrix spikes, and laboratory duplicates)

e. If surrogates are required by the method, are their recoveries within the control limits in the spiked blank

f. Is the recovery of surrogates within control limits in the samples, and if not, has the sample preparation and analysis been repeated, and have the recoveries been acceptable in the repeated analysis

g. Is the recovery of spiked compounds in the laboratory spiked blank acceptable

h. Is the recovery of spiked compounds in the spiked sample acceptable and if not, has there been an acceptable explanation or a repeat of the analysis

i. Do duplicate analyses in the run sequence exhibit precision within the control limits

j. Is the documentation in order

If the answers to all the questions above is "yes", the QA/QC personnel will initial the review checklist and date it, and submit the form to the data management thereby releasing the batch to be reported.

If the answer to any of the questions is negative, corrective action will be taken initiated by the QA/QC in association with the project manager for those projects that have a specifically assigned project manager. In essence, the QA/QC personnel checks the batch for completeness, accuracy, and precision. If all criteria are met, the batch is released. If some are not met, the batch may still be released, depending upon what criteria are not met and what is the explanation for not meeting the criteria. There are certain circumstances when the release of the data is not allowed regardless of the rationale for the lack of acceptability. These are:

1. Continuing calibration was either not performed or did not meet acceptance criteria

2. Laboratory control standards (spiked blanks) were not run or did not meet acceptance criteria

3. Data sets are not complete

Corrective action for these situations will be discussed in Section 7.0.

If certain aspects of the data do not meet acceptance criteria, but consultation with the project manager or the laboratory manager resulted in a decision to release the data, the QA/QC Section Manager will annotate on the checklist the shortcomings of the data, the decision to proceed with reporting the data, and the person with whom that decision had been reached. The form will then be submitted to the Data Management Section so that the final report can be prepared.

If the decision has been made not to release the data, the form will be marked DO NOT RELEASE DATA, and submitted to the Data Management Section.

In addition to validating the data based on batch runs, it is still necessary to validate specific jobs before they are released. Specifically, releasing a job consist of insuring that all parameters have been analyzed for, and are reported. The results for each of the parameters have the QA/QC release through approval of the individual batches that make up all the parameters associated with the job. Documentation for the job is complete. It

is the responsibility of the Data Management group to assure that all data are validated and entered, and that the documentation is complete. QA/QC will only spot check the final reports.

6.4 DATA COMPILATION

Upon receiving the validation checklist, the Data Management Section personnel will review the job file to confirm that all the data are in the file, and references are made to those items which are associated with the job but are present in other files.

The data will then be entered in standardized forms, and the report printed out.

6.5 FINAL REVIEW

When the final report has been printed, another person, not the one entering the data, will proof the report against the raw data in the file. If all the data appear properly entered, the report will be sent to the client. A copy of the report will be placed in the job file, and the file will be transferred to the storage of completed jobs.

A form will also be generated daily, showing which jobs have been reported on that day. That form will be submitted to the Sample Management Section, so that they can schedule the disposal of the samples.

7.0 CORRECTIVE ACTION

There are many areas of the laboratory functions which may require corrective action. The decision to undertake corrective action, and the ensuing action must be documented so that traceability can be maintained. The point of originating the corrective action varies, depending upon the mode of detecting that such action is necessary. It is, however, frequently the role of the QA/QC Section to initiate such action, simply because it is this section which is most exposed to the malfunctions of the laboratory as they reflect upon the data produced, in historical perspective. Those actions that affect the quality of the data will be recorded and the record maintained by the QA/QC Section.

7.1 IDENTIFICATION OF POTENTIAL PROBLEM

Identification of sources of problems is not always an easy matter. It is, therefore, not expected that the QA/QC Section be able to identify the source of the problem when it is detected through data validation. The QA/QC Section will be responsible for informing the Section Manager and the Laboratory Director that a problem appears to exist in a particular type of analysis, and the data of that type of analysis will not be accepted until the problem is isolated and corrected. It will be the responsibility of the Section Manager to address the identification of the source of the problem, and the responsibility of the Laboratory Director to assure that the Section Manager is acting upon the need for corrective action.

In some situations, the need to correct an operation is apparent to the analyst, and does not originate from the data validation process. For example, instrumental failures are determined by the analyst, and the corrective action is taken in the form of repairing the instrument either through a service call or through the laboratory personnel. Such action must be recorded in the instrument maintenance log, and QA/QC will be informed of such action, so that close scrutiny can be paid to the analysis just preceding the instrumental failure. If these analyses met the required acceptance criteria, no further action will be taken relative to that data.

In other situations, the time of data validation is the more logical time to isolate a potential problem. For example, if a systematic drift occurs due to chromatographic

column contamination, it is easier to identify at the time of validating the data and comparing the results with historically available information.

7.2 PROBLEMS AND ACTIONS

7.2.1 Continuing Calibration Outside Acceptance Range

When the continuing calibration is outside the acceptance range, the problem should be identified by the analyst and corrected before further sample processing is undertaken. On some occasions, the nonacceptability of the continuing calibration will not be caught by the analyst. In these cases, QA/QC will notify the responsible section that a new initial calibration curve must be prepared.

The data on all samples that have been analyzed following the last time that the calibration was within specification will be rejected by the analyst, the Section Manager, or the QA/QC Section, depending upon at what stage the nonacceptability of the calibration curve was determined. QA/QC Section will be notified immediately, using the Form shown in Figure 5-2. The samples will be reanalyzed after the new initial calibration curve has been constructed.

7.2.2 Calibration Standards Exceeding the Permitted Holding Time

If calibrations standards have been continuously used beyond their permitted shelf-life, the Section responsible for the analysis will be notified. The section will be responsible for preparing fresh calibration standards, and the instrument calibration will be checked against the new standards. If the previous runs, performed with the expired standards meet the acceptance criteria based on the new standard, the data generated will be considered valid, in spite of the use of an expired calibration standard.

If the calibrations performed with the expired standard do not meet the acceptance criteria, when measured against the new standards, the samples that have been analyzed against the expired standard will be reanalyzed.

7.2.3 Laboratory Method Blanks Exceed Method Detection Limit but are Below Quantitation Limit

When laboratory blanks exhibit the presence of target analytes at a level exceeding the method detection limit, but still below the quantitation limit, the responsible Section will be notified.

The responsible section will check the reagent blanks that have been retained at the time of use of the reagents, in order to determine if contamination or interferences are due to impurities in the reagents. If this is the case, the reagent batch will be discarded, and new reagents, from fresh containers will be used.

If the reagents appear to be sufficiently pure, the cleanliness in the laboratory will be reinforced to establish if the source of problems may have been contamination of the apparatus.

The data for samples associated with the blank will be accepted; however, it will be blank corrected, except for projects where such correction is not allowed.

7.2.4 Laboratory Method Blank Exceeds the Quantitation Limit

When the laboratory method blank exceeds the quantitation limit, the Section responsible for the analysis will be notified. As discussed in Section 7.2.3, the analysts will check for potential contamination of reagents and apparatus. If the reagents are contaminated, the existing batch will be rejected, and a fresh batch, from new containers will be prepared..

If the problem arose from the apparatus, whether glassware or instrumental, the problem will be corrected within the analytical section, and the correction documented before any further analyses can be undertaken. Notification of the corrective action will be submitted by the Section Manager to the QA/QC Section.

The data associated with the failed method blank will not be accepted. The samples will be reanalyzed to produce acceptable data.

7.2.5 Laboratory Control Standard Exhibits Recoveries Outside the Acceptance Criteria

When the laboratory control standard (spiked blank) does not meet the acceptance criteria, the batch of samples associated with the laboratory control standard will be reanalyzed. The original data will be rejected. In methods that require the use of a laboratory control standard at frequent intervals through the day, only those samples analyzed since the last acceptable laboratory control standard analysis will require repeating.

Before repeating a whole set of preparations of samples, the calibration of the instrument or method shall be checked. If the instrument is within calibration, the samples will require repreparation. If the instrument calibration has drifted, the prepared samples from the initial preparation can be reanalyzed after the instrument has been recalibrated.

7.2.6 Surrogates and Sample Spikes Exhibit Recoveries Outside the Acceptance Limits

When recoveries from spiked samples are outside the acceptance limits, but the laboratory spiked blank is within the acceptance criteria, the poor recovery or enhanced apparent recovery may be due to matrix effect. One such sample from the batch will be reprepared and reanalyzed. If the same phenomenon is observed, it will be assumed that the failure to meet recovery criteria was in fact a matrix effect. This information will be included in the report to the client: however, the original data will be accepted.

7.2.7 Control Chart Exhibits a Regular Trend

By their very nature, the individual points that make up the control chart for any analyte vary randomly about a mean value. The control chart is used to assess the acceptability of recovery data on the basis of historical data. The

control chart is also used to warn the analyst that some consistent problem or deviation in the method may be occurring.

When five successive points on the control chart form a steady pattern, either regularly increasing or regularly decreasing, they imply that some change is occurring in the analytical scheme. Even if all the points are within the control limits, a warning will be issued by the QA/QC Section to the responsible section of the laboratory to investigate the cause of the pattern. If in fact a change has occurred in the method, and if the change indicates an improvement in recoveries (an improvement is defined as approaching complete recovery, not necessarily an upward trend), then a new control chart will be established, and subsequent data will be compared to the new control chart limits. If a change has occurred that worsens the recovery, it will be the responsibility of the Section Manager to assure that a return to the previously used technique is made in his section.

7.2.8 Poor Performance on an Internal System Audit or an External Performance Evaluation

Internal system audits are quarterly samples issued by the QA/QC Section, using known spiked solutions to determine the performance in every section of the laboratory. External performance evaluation is either done through a contract required performance audit, or through voluntary participation in interlaboratory studies.

When the achieved results on these audits fall below acceptable standards, as defined on the basis of historical recovery data, a thorough review of the system will be initiated. The QA/QC Section is responsible for the initiation of the process. The first step will consist of complete review of the correctness of the documenting of the job, and the calculations of the results. This process will be performed by the personnel of the QA/QC Section. When the results of the review are complete, a memo will be issued to the responsible section of the laboratory, itemizing the deficiencies that have been identified. If no deficiencies have been identified, the difficulty may be with the performance of the analyst with the analytical method, or incorrect preparation of the audit sample.

The manager of the analytical section will be responsible for investigating the source of the problem if it is internal to the section. His findings and the corrective action taken will be reported to the manager of the QA/QC Section. If the findings are negative (the system has functioned correctly), the QA/QC Section will issue a new performance evaluation audit sample, prepared from fresh standards, preferably from a different source than the first one submitted. If the analysis of the new audit sample is found to be within acceptance limits, it will be assumed that the prior problem was with the audit sample itself.

APPENDIX B

RI/FS
HEALTH AND SAFETY PLAN
TYLER REFRIGERATION PIT
SUPERFUND SITE
SMYRNA, DELAWARE

Prepared For:
Clark Equipment Company
100 N.Michigan Street
South Bend, IN 46634

Prepared By:
Environmental Resources Management, Inc.
855 Springdale Drive
Exton, Pennsylvania 19341

File No.: C27-01-01

The ERM Group

HEALTH AND SAFETY PLAN
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
TYLER REFRIGERATION PIT SUPERFUND SITE
SMYRNA, DELAWARE

12 November 1991

David Steele
ERM Project Manager

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The ERM Group

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SECTION 1
INTRODUCTION

This Health and Safety Plan (HASP) has been developed for the Tyler Refrigeration Pit Superfund Site (hereinafter referred to as the "Site") investigation activities, and is intended to provide recommended Health and Safety procedures for ERM employees and ERM subcontractor employees participating in the on-site field activities as part of the investigation.

Procedures and protocols set forth in this plan are designed to reduce the risks of exposure to chemical substances and materials that may be present in the soils, sediments, ground water, air or surface water at the facility. The procedures contained herein were developed in accordance with the provisions set forth by 29 CFR 1910.120 (Hazardous Waste Operations and Emergency Response), and include protocols dictated by ERM, Inc. in accordance with ERM's experience in similar field investigations.

ERM, Inc. shall be responsible for ensuring that this Health and Safety Plan is properly implemented, and that all site activities conducted during the investigation are in compliance with the provisions indicated in this plan. The recommended procedures and protocols outlined in this document may be modified during the course of the investigation as additional information is made available during on-site characterization or from laboratory chemical analyses. These modifications will be issued in the form of revisions to specific pages or sections of the Health and Safety Plan. A revised table of contents will also be issued for verification of updated pages.

This plan has been developed to ensure that all field activities and site operations associated with the investigation are in accordance with Federal guidelines as established by the following guidance documents:

Government Regulations	Topic
29 CFR 1910.120	Hazardous Waste Site Operations
29 CFR 1910.20	Recordkeeping/Recording
29 CFR 1904	Recordkeeping/Recording
29 CFR 1910.1000	OSHA Permissible Exposure Limit
29 CFR 1926	Construction Activities

Site-specific investigative activities will also employ procedures and protocols as outlined in ERM, Inc. Standard Operating Procedure for the Collection of Environmental Samples (September 1989), with particular emphasis on personnel training requirements and maintenance and calibration of field equipment.

SECTION 2
PROJECT PERSONNEL AND RESPONSIBILITIES

The following responsibilities and authorities have been assigned to designated personnel for the investigation field activities at the Site.

ERM, Inc. Project Director

Alan Funk is the Project Director for the investigation. The Project Director is responsible for overall technical oversight and coordination of the various components of the investigation. The Project Director will also interface directly with the Project Manager and Task Managers to perform an overall review of project activities and assess technical performance.

ERM, Inc. Project Manager

David Steele is the Project Manager for the investigation. He will act in a supervisory capacity over all ERM employees and subcontractors with respect to ERM's contractual obligations to Clark Equipment Company, and will be primary point-of-contact with USEPA management and technical representatives. Mr. Steele is responsible for assuring that this Health and Safety Plan is adhered to, and that all project activities conform to guidelines and protocols contained in this document.

ERM, Inc. Site Operations Manager

Edward Sullivan is the Site Operations Manager for the investigation. He will be under the direct supervision of ERM's Project Manager to oversee the performance of field investigative activities and to ensure the timely completion of related investigation technical tasks. The Site Operations Manager is directly responsible for ensuring that the Site Safety Officer has correctly implemented the HASP, and that any subcontractors to ERM are performing in accordance with the HASP.

ERM, Inc. Health and Safety Coordinator

Mr. Robert Deist is the Health and Safety Coordinator (HSC) for the investigation. Mr. Deist has a sound working knowledge of state and federal occupational and health regulations. He also has had formal training and work experience in safety-related work at hazardous waste sites. As HSC, he is responsible for insuring that all project personnel are aware of the guidelines set forth in this Health and Safety Plan, including inherent risks of chemical exposure associated with work of this nature, and are properly trained in the use of

advanced safety equipment and protective clothing designed to protect against chemical exposure.

ERM, Inc. Site Safety Officer

Site Safety Officer will be designated per task. He will be directly responsible for ensuring compliance to the HASP by all ERM employees and subcontractors to ERM (drilling and surveying subcontractors). He will also have complete authority (during the performance of field tasks on-site) to enforce the HASP, and immediately cease field operations if violations of the HASP are committed by field personnel or subcontractor personnel.

ERM's Site Safety Officer (SSO) is an experienced professional with extensive knowledge of state and federal occupational health and safety regulations. The SSO will be responsible for performance of air monitoring in the workspace to ensure proper levels of personnel protection, and will maintain Site Health and Safety records in the project field book which will contain all relevant Health and Safety data. The SSO will perform the necessary calibration of and will ensure the necessary maintenance is accomplished on all instruments used in the protection of personnel health and safety.

If site conditions permit, the SSO has the prerogative to appoint the senior-most ERM employee on the Site as the field SSO. The field SSO will be responsible for ensuring all health and safety procedures are followed and documentation accomplished in accordance with the Health and Safety Plan and the directives of the SSO.

SECTION 3
SITE SPECIFIC INFORMATION

The Site is a former lagoon area located at 655 Glenwood Ave, Smyrna, Delaware. The Site is situated on a parcel of property that is currently occupied by Metal Masters Food Service Equipment Company, Inc. (Metal Masters), but was formerly owned by the Tyler Refrigeration Corporation. The Site is approximately 1/2 mile southwest of the center of the town of Smyrna (population 4750).

The Site consists of an area which formerly contained two wastewater lagoons (or pits). The Site is located in the northeast portion of the Metal Masters property. Aerial photographs suggest that the northernmost lagoon was approximately 70 feet x 70 feet in size and the southernmost lagoon was approximately 60 feet x 60 feet. The lagoons apparently received wastewater from manufacturing operations at the property. Based on a review of aerial photographs, the two lagoons were present on the property from as early as July 1954. This review also indicates that sometime between 1973 and 1975, the contents of the lagoons were excavated and removed. The lagoons were subsequently backfilled and regraded. The Site is currently covered by a lawn and an asphalt parking lot for the manufacturing building located on the property.

Little is known regarding the uses of the Site and surrounding property prior to 1946. Beginning in 1949, a plant was operated on the property to manufacture refrigerators. In 1951, Tyler Refrigeration Corporation (Tyler) assumed control of the refrigeration manufacturing operations at the property. According to aerial photographs, sometime prior to July 1954, two lagoons were constructed in the northeast portion of the property. In 1963, Tyler transferred the property to Clark Equipment Company (Clark) as part of a transaction whereby Tyler became a part of the refrigeration division of Clark. Clark manufactured refrigeration-related equipment at the property until approximately 1976.

According to NUS ("A Field Report for Tyler Refrigeration" 1986), wastewater discharges from manufacturing process were connected to a municipal sewage system in 1969. In addition, aerial photographs indicate that sometime between 1973 and 1975, the contents of the lagoons were excavated and removed. Lagoon materials and or soils were reportedly removed to a depth of approximately 20 feet.

In 1978, the property was purchased from Clark by Metal-Masters. Metal Masters has been manufacturing food service equipment at the property since 1978.

In 1982, EPA commissioned Ecology and Environment, Inc. ("E&E") to perform a Preliminary Assessment/Site Investigation in connection with the Site. 1,1,1-trichloroethane (TCA) and 1,1-dichloroethane (DCA) were detected in one of the soil samples collected as a part of this study at concentrations of 15 micrograms per kilogram (µg/Kg or parts per billion) and 10 µg/Kg respectively. Toluene was detected in a second soil sample at a concentration of 25 µg/Kg.

In 1988, EPA commissioned the Delaware Department of Natural Resources and Environmental Conservation (DNREC) to conduct a further investigation at the Site. Monitoring well nests were installed at three locations and sampled. 1,1,1-TCA was detected in each of the three shallow wells at concentrations ranging from 5 to 110 micrograms per liter (µg/L), 1,1-DCE was detected in well S-1 at a concentration of 8 µg/L, and chromium (total) was detected in wells S-2 and D-2 at concentrations of 19 and 113 µg/L, respectively. These substances are very near or well below the maximum contaminant levels (MCLs) set under the Safe Drinking Water Act.

EPA added the Site to the National Priorities List (NPL) in February 1990. Although several Potential Responsible Parties (PRPs) have been identified by EPA, Clark has agreed to enter into an Administrative Order on Consent (AOC) with EPA to conduct a RI/FS for the Site.

SECTION 4
SUMMARY OF PROPOSED REMEDIAL INVESTIGATION FIELD TASKS

The following items are included among the proposed field tasks to be performed during implementation of the Work Plan. These items are discussed in more detail in Sections 2 and 3 of the Work Plan:

Investigative Task	Task Descriptions
Soil Borings and Surface Soil Samples	- Advance soil borings to the water table using a hollow stem augers, hand auger to depth of 1 foot for surface soil sampling..
	- Collect soil samples (split spoon samples) from depth intervals as listed in the Work Plan for laboratory analyses.
Installation of Monitoring Wells	- Install monitoring wells. - Develop each monitoring well
Ground Water Sampling	- Purge monitoring wells and collect ground water samples.
Synoptic Water Level Measurements	- Open monitoring wells and allow to vent. - Collect water level measurements in each well.
Well and Sample Location Survey	- Survey horizontal and vertical positions of all sample and monitoring well locations.

SECTION 5
HAZARD ASSESSMENT

Previous on-site soil samples have indicated the presence of several volatile organic compounds.

Table 5-1 presents a listing of chemical compounds that are believed to be potentially present on-site based upon previous chemical analyses. Permissible Exposure Limits (PELs) presented in this table were derived from OSHA's 1989 PEL Final Rule.

Table 5-2 presents an example listing of some of the substances that may be encountered during the course of field activities. The substances listed have been chosen for informational purposes, and are intended to provide a framework for the development of relevant exposure information that is easily recognizable by field personnel and the Site Safety Officer. This list is not all encompassing, but is meant to serve as a guide for assessing exposure potential, possible routes of exposure, symptoms of overexposure, and relevant chemical-specific and physiologic information.

Table 5-3 presents a listing of potential physical hazards that may be encountered during the course of implementing the investigation activities.

TABLE 5-1

POTENTIAL ON-SITE SUBSTANCES
TYLER REFRIGERATION PIT SUPERFUND SITE

Constituent	PEL*	
Toluene	100	ppm
Chromium (Metal)	0.5	mg/m^3
1,1-Dichloroethene	5.0	ppm
1,1,1-Trichloroethane	350	ppm
1,1-Dichloroethane	100	ppm

*Time weighted average (TWA) average concentration for a normal 8-hour work day and a 40-hour work week.

TABLE 5-2
SELECTED POTENTIAL ON-SITE SUBSTANCESS AND ASSOCIATED EXPOSURE INFORMATION
Tyler Refrigeration Pit Superfund Site

COMPOUND	CAS	TVL/PEL	CHARACTERISTICS	ROUTE OF EXPOSURE	SYMPTOMS OF OVEREXPOSURE*	TARGET ORGANS
Toluene	108-88-3	100 ppm	Colorless liquid; Aromatic odor	Inhalation Absorption Ingestion Contact	(1), (2), (3), (4)	Blood, CNS, skin, eyes liver, urinary tract
Chromium	7440-47-3	0.5 mg/m3	Blue-white to steel gray, lustrous, brittle hard solid	Inhalation Ingestion	(1)	Respiratory System
1,1-Dichloroethene	75-35-4	5 ppm	Colorless gas or liquid with a pleasant odor at high concentrations.	Inhalation	(1)	Liver, Blood, CNS, Respiratory System, Lymphatic System
1,1,1-Trichloroethane	71-55-6	350 ppm	Colorless liquid with a mild chloroform-like odor.	Inhalation Ingestion Contact	(1), (2), (3), (4), (5)	Skin, CNS, CVS, eyes
1,1-Dichloroethane	75-34-3	100 ppm	Colorless, oily liquid with a chloroform-like odor.	Inhalation Ingestion Contact	(1), (4)	Skin, Liver, Kidney

(1) Eye, nose, throat, skin irritation or burns
(2) Headache, fatigue, nausea
(3) Lightheaded, some nausea, dull visual and audio response
(4) Central nervous system disorder, convulsions, sweating
(5) Potential or known carcinogen
* PEL - Permissible Exposure Limits (OSHA) - 8 hour exposure
TLV - Threshold Limit Value (AGCIH) - 8 hour exposure

TABLE 5-3
POTENTIAL HEALTH AND SAFETY PHYSICAL HAZARDS
TYLER REFRIGERATION PIT SUPERFUND SITE

POTENTIAL HAZARD*	DESCRIPTION	LOCATION	PROCEDURE USED TO MONITOR/REDUCE POTENTIAL HAZARD
Heavy Equipment	Drill Rigs, Machinery	Throughout Site	Personnel maintain eye contact with operators; hard hats, safety shoes, and eye protection worn (as appropriate) during equipment operation.
Refuse and Materials	Construction refuse and construction materials	Throughout Site	Maintain clean work areas; dispose of refuse immediately; do not block access routes with materials.
Heat Producing/ Electrical Equipment	Generators/Drill Rigs	Throughout Site	Operate equipment away from vegetation and other materials that may ignite. Maintain fire-fighting equipment in the vicinity of operating equipment.
Heat Stress/Cold Exposure	Personnel working under extreme temperature are subject to adverse temperature-related effects	Throughout Site	Employ buddy system. Each worker is responsible for visually monitoring his/her partner for signs of heat stress/cold exposure. Site safety personnel will also monitor worker's conditions and establish work/rest regimens and recommend appropriate diet.
Chemical Exposure	Personnel can be exposed to various compounds associated with the site	Throughout Site	Follow guidelines in Safety Plan. Be familiar with signs and symptoms of exposure and first aid procedures. Report suspected over-exposure to supervisor immediately.

*Based on equipment or situations that can cause a hazard.

SECTION 6
ACTION LEVELS FOR PERSONNEL PROTECTION

Specific action levels have been established for personnel involved in field activities. These action levels apply to all ERM, Inc. and ERM, Inc. subcontractor personnel. The action levels are to be observed by all personnel in all areas of the Site. The action levels presented below shall apply to site-related activities, and are to be observed by the Site Safety Officer (SSO) when determining the need to upgrade the required level of personnel protective equipment.

Action Levels for Personnel Protection*
With presence of 1,1-Dichloroethene (DCE)
confirmed with detector tubes

Total Volatile Organic Compounds (ppm)*	Required Level of Personnel Protection
Background VOC Level in ambient air to 3 ppm	Level D protection, with respiratory protection readily available to all personnel.
3 ppm to 30 ppm *if DCE present*	Level C protection, with half-face-respirator equipped with cartridges *for organic vapors, dust, fumes, mist, asbestos, and radionuclides*
30 to 150 ppm if DCE present	Level C protection, with full-face respirator equipped with cartridges *for organic vapors, dust, fumes, mist, asbestos, and radionuclides*
Greater than 150 ppm *if DCE present*	Level B protection, with self contained breathing apparatus, SCBA and Grade D or better breathing air supply.

* Action levels based upon total VOC measured in the breathing space using an OVA, and are based on approximately *one half* the PEL for 1,1-Dichloroethene.

Action Levels for Personnel Protection*
With DCE not present

*Total Volatile Organic Compounds (ppm)***	*Required Level of Personnel Protection*
3 ppm to 50 ppm DCE not present	*Level D protection, with respiratory protection readily available to all personnel.*
50 ppm to 500 ppm DCE not present	*Level C protection, with half-face-respirator equipped with cartridges for organic vapors, dust, fumes, mist, asbestos, and radionuclides*
500 ppm to 1000 ppm DCE not present	Level C protection, with full-face respirator equipped with cartridges *for organic vapors, dust, fumes, mist, asbestos, and radionuclides*
>1000 ppm	Level B protection, with self contained breathing apparatus, SCBA and Grade D or better breathing air supply.

** Action levels based upon total VOC measured in the breathing space using an OVA, and are based on *one half* the PEL for 1,1-Dichloroethane and *Toluene*.

SECTION 7
PERSONNEL PROTECTIVE EQUIPMENT

The level of protection to be worn by field personnel and subcontractors will be defined and controlled by the Site Safety Officer (SSO). Basic levels of protection for general site operations are outlined in Section 8. When more than one hazard area is indicated during site operations, further delineation of the hazards shall be provided by the SSO after a complete review of operational requirements, operational conditions, and or monitoring at the particular operations being conducted. Protection may only be upgraded/downgraded after receiving authorization from the SSO. More detail on Personal Protective Equipment (PPE) may be found in Attachment A.

SECTION 8
LEVELS OF PERSONNEL PROTECTIVE EQUIPMENT FOR INVESTIGATION ACTIVITIES

This HASP is intended to provide appropriate protection to these individuals during planned investigative activities, decontamination of personnel and equipment, site layout, and general preparatory activities, etc. The HASP has been designed to allow for upgrading or downgrading the level of *Personnel Protective Equipment (PPE)* protection to conservatively preclude any potential for human exposure. Again, the Site Safety Officer is solely responsible for determining the levels of PPE to be used during on-site activities, and has the option to upgrade the level of PPE protection based on air monitoring results *or other factors such as splashing or excessive contact with contaminated site media.*

Site Activity	Location	PPE Required
Surface Soil and Subsurface Borings (Drilling Subcontractor)	See Work Plan	Level D for intrusive activities; Option to upgrade to Level C as approved by SSO
Monitoring Well Installation (Drilling Subcontractor)	See Work Plan	Level D or as determined by SSO
Monitoring Well Development	See Work Plan	Level D or as determined by SSO
Surveying Activities (Survey Subcontractor)	All Sampling and Monitoring Well Locations	Level D, contingency to upgrade to Level C when wells open.

Basic emergency equipment (i.e. first aid kits, fire extinguishers, etc.) will also be available *in all ERM vehicles* during the performance of all investigation activities.

SECTION 9
MEDICAL MONITORING REQUIREMENTS

All ERM employees, as well as subcontractor employees under the supervision of ERM personnel, are required to have been under a medical surveillance program provided by a licensed physician practicing in the fields of occupational health or environmental health. At a minimum, each individual shall receive an annual Health and Safety physical examination to include the following testing:

- Medical History and Physical, including:
  - Medical questionnaire
  - Completion of medical history with occupational risk factor analysis
  - Examination by physician
  - Evaluation of test results
  - Brief report sent to employer covering specific requested areas as well as pertinent positive findings; report sent to family physician and employee by request
- Pulmonary Function Testing (FEV_1, FVC)
- EKG (12-lead)
- Lab tests, including:
  - Urinalysis
  - Blood "Chemzyme" Analysis (Chem 18)
  - Coronary Risk Screen
  - Complete Blood Count with differential
- Audiometric testing - supervised by board-certified staff otolaryngologist
- Visual Acuity and Tonometry - supervised by board-certified staff ophthalmologist

ERM has maintained an accurate file of all medical records and will record personnel exposure monitoring data as required by Subpart C of 24 CFR 1910.120 of the Occupational Safety and Health

Administration. These files are located in our Exton, Pennsylvania office and can be made available for authorized individuals only to review by contacting ERM,Inc.'s Corporate Health and Safety Program Manager, Mr. Bob Deist, at (215) 524-3775.

Subcontractor personnel will be required to document their compliance with the applicable Medical Surveillance program requirements by completing the "Subcontractor Occupational Safety and Health Certification" form, presented as Exhibit 9-1. A completed Exhibit 9-1 given to the SSO will be required prior to subcontractor personnel going on site.

Additionally, the text presented in Attachment B provides additional general information regarding the ERM, Inc. Medical Monitoring program.

EXHIBIT 9-1

SUBCONTRACTOR OCCUPATIONAL SAFETY AND HEALTH CERTIFICATION

PROJECT: _____

SUBCONTRACTOR: _____

1. Contractor certifies that the following personnel, to be employed during the Tyler Refrigeration RFI, have met the following requirements of the OSHA Hazardous Waste Operations Standard (29 CFR 1910.120) and other applicable OHSA standards, as required by ERM.

Subcontractor Personnel	Training	Respirator Certification	Medical Exam
e.g. John Doe	1/2/89 ERM	MSA med (1/89)	2/1/89 Paoli

2. Subcontractor certifies that it has received a copy of the Site Safety and Health Plan and will ensure that its employees are informed and will comply with its requirements.

3. Subcontractor further certifies that it has read and understands and will comply with all provisions of its contractual agreement.

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SECTION 10

REQUIRED HEALTH AND SAFETY PROGRAM

At a minimum, all applicable employees must meet the training requirements specified in 20 CFR 1910.120 by having been trained in the areas listed below. ERM contractors and subcontractors must acknowledge their compliance to the training requirements by completing the form previously presented as Exhibit 9-1.

- Site Safety Officer and Site Management Responsibilities - personnel must understand Site Safety Officer and Site Management responsibilities and authority.
- Site-Specific Health and Safety Hazards - personnel must be informed of specific potential hazards related to site and site operations.
- Personnel Protection Equipment (PPE) - personnel must be trained in proper used of personal protective equipment.
- Safe Work Practices/Engineering Controls - personnel must be informed of appropriate work practices and engineering controls that will reduce the risk of exposure to potential site hazards.
- Safety Equipment Use- personnel must understand the use of monitoring instruments and other safety equipment.
- Medical Surveillance Program - personnel must be informed of requirements for medical surveillance of hazardous waste site employees.
- Site Control Methods - personnel must understand site methods used to reduce exposure to on-site and off-site personnel, and must observe the established safety zones on-site.
- Decontamination Procedures - personnel must be trained in proper decontamination operations and procedures for personnel.
- Emergency Response - personnel must be trained in proper emergency response operations and procedures, and will be able to access associated emergency contact numbers.
- Confined Space Entry/Special Hazards - personnel involved in specific hazardous activities, such as confined space entry and drum handling, must receive training in appropriate techniques to employ during such operations.

Subcontractor personnel will document their compliance with training and medical program requirements by completing the "Subcontractor Occupational Safety and Health Certification form", Exhibit 9-1.

All ERM and subcontractor personnel will attend a safety and logistics briefing by the Site Safety Officer before the commencement of field activities.

A more detailed discussion of training requirements and areas of specialized training are included in Attachment C of this HASP.

SECTION 11
HEALTH AND SAFETY PROTOCOLS DURING SITE OPERATIONS

11.1 Air Monitoring Program

All proposed site activities will involve monitoring of the ambient airspace in the work area by the SSO (or a qualified designee, if there are site activities being performed concurrently). A flame ionization detector (FID) or a photo ionization detector (PID) will be used to monitor total VOC concentrations (in methane or isobutylene equivalents respectively). At the initiation of each work activity or work period, the SSO (or qualified designee) will measure and record the background levels of total VOCs in the ambient airspace. Additionally, relevant meteorologic data will be estimated and recorded in the project field book, with particular emphasis on wind speed, wind direction, relative humidity, and ambient air temperature. The potential for volatilization of VOCs will be assessed based upon the activity to be performed (intrusive versus non-intrusive), and the meteorologic conditions existing at the time the activity is to take place.

Air Monitoring During Drilling Operations

Air monitoring will be performed at each drilling location during the performance of soil borings, well installation, well development, and subsurface soil sampling operations. A FID (Century 128 OVA) or a PID (Photovac Tip II, Microtip, or TVM) will be utilized to monitor the breathing zone in the workspace surrounding the well location, within the annular space of the borehole, and from all geologic soil samples examined upon their retrieval. A combustible gas/LEL/O_2 meter (MSA 260 or equivalent) equipped with an alarm may, if directed by the SSO, be used to monitor the borehole and annulus for the presence of any accumulated combustible gases. Similar monitoring of any fluids generated during well installation/development will also be conducted.

Air Monitoring During Other Site Operations

The need to perform air monitoring during other site activities shall be determined by the SSO. However, air monitoring during survey activities will be conducted.

11.2 Site Safety Zones

Site safety zones will established for the performance of field activities during the investigation. Location of these proposed site safety zones will be determined by the SSO based on air monitoring. These locations may be subject to modification by the SSO dependent upon

the type of investigative activity proposed, levels of contaminants encountered, etc.

Exclusion Zone - The area immediately surrounding the sampling or well installation activity, shall be considered the Exclusion Zone (EZ). All entry/egress from this area shall be through a single entry/exit point established in an upwind direction from the proposed activity. The EZ shall not be accessed from any other direction or location without explicit approval of the SSO, or unless an emergency situation has developed which necessitates immediate evacuation of the work area (contaminant release, fire, explosion, etc). The proper levels of PPE shall be worn at all times, as specified by the SSO, within the EZ. Only authorized personnel are permitted in the EZ.

Contaminant Reduction Zone - A primary Contaminant Reduction Zone (CRZ), designed to ensure the proper decontamination of all equipment entering and leaving the primary areas of investigation, will be established. A decontamination pad for heavy equipment (drill rigs, backhoes, etc.) and sampling equipment decontamination area will be established on the parking lots over the former lagoons.. Materials storage (well casing, screen, drilling supplies, etc.) will not be permitted within the CRZ. Other equipment decontamination activities (including sampling equipment decontamination) shall be performed in a separate portion of the CRZ. Sample containers and sampling equipment (other than that associated with truck mounted drill rigs) shall be stored within the CRZ. Additionally, the proper levels of PPE shall be worn at all times, as specified by the SSO, within the primary CRZ.

Support Zone - The Support Zone (SZ) will be established and marked, and will consist of support vehicles, emergency communication equipment, first aid supplies, and other equipment needed to monitor or perform site sampling activities. This area shall remain as the "clean" area due to strict enforcement decontamination procedures by the SSO.

11.3 Site Communications

Walkie-Talkies - Hand held units shall be utilized as needed by field teams for communication between downrange operations and the SSO and Support Zone.

Telephones - A pay telephone is located in the Metal Masters building at the employees' entrance for communication with emergency support services/facilities.

Hand Signals - To be employed by downrange field teams, along with utilizing the buddy system. These signals are also very important when working with heavy

equipment where audio contact may be difficult or impossible.

Hand Signals shall be known by the entire field team before operations commence and will be covered during site-specific training and briefings.

Hand signals to be used - and their meanings:

- Hand gripping throat - out of air; cannot breathe;
- Grip partners wrists or place both hands around waist - leave immediately;
- Hands on top of head - need assistance;
- Thumbs up - ok; I am alright; I understand;
- Thumbs down - no; negative.

11.4 Site Access/Site Control

Access to the active investigation work areas will be limited to only trained and authorized personnel, to include: ERM employees, subcontractor personnel, State and Federal regulatory agency personnel, and designated client representatives. All persons who do enter the immediate work area(s) will be required to follow sign-in procedures, as needed. Each worker is responsible to sign, indicate the time in and out, and indicate the intended work area on the Authorized Site Personnel List, held by the SSO or his designee. The Site Operations Managers will be responsible for ensuring that this sign-in procedure is implemented and documented correctly in the site logbooks.

Access into established zones, including the Exclusion Zone (EZ) and Contaminant Reduction Zone (CRZ) will be limited to only those personnel wearing the appropriate PPE.

Site Control considerations dictate that specific procedures be followed to ensure adequate site control so that persons, who may be unaware of existing site conditions, are informed of the possibility of being exposed to inherent site hazards. All heavy equipment and drill rigs shall be stored in a secured area upon the completion of each day's activities. Any excavations left unfilled and unattended by project personnel will be appropriately barricaded and visibly posted with the appropriate warning signs, temporary fencing, or necessary access restrictions and safety precautions. Additionally, all potentially contaminated materials generated during performance of the investigation activities will be containerized and placed in a secure

area, and all monitoring wells locked and secured to prevent unauthorized access or tampering.

11.5 Health and Safety Violation Policy

Health and safety procedures have been established to protect site personnel and to prevent the spread of substances of concern.. Therefore, it is imperative that all personnel adhere to the procedures outlined in this health and safety plan and those issued by the SSO. Because of the potentially grave consequences as a result of personnel not complying with the health and safety procedures, a worker dismissal policy has been established.

ERM employees and subcontractor employees are subject to site dismissal by the SSO or his designated representative based on three health and safety violations of the same nature. The procedure, outlined below, is applicable to all site personnel, regardless of position.

1st violation: The worker is verbally instructed in the proper procedure and the offense is noted in the health and safety file. The worker's foreman or immediate supervisor is given written notice of these actions within 24 hours.

2nd violation: The worker is verbally instructed in the proper procedure and warned that the next offense will constitute grounds for dismissal. The offense and warning are noted in the health and safety file. The worker's foreman or immediate supervisor will be given written notice of these actions within 24 hours.

3rd violation: The worker is given verbal and written instruction to depart the site following proper termination procedures (i.e. the turning in of company gear, reporting to foreman, setting up exit physical, etc.). The violation and termination action is noted in the health and safety file. The worker's foreman or immediate supervisor will receive immediate verbal notice and written notice within 24 hours of the dismissal.

If an employee wishes to contest a violation ruling, the employee shall do so through his/her foreman or immediate supervisor. The SSO or designated representative shall consider all points and either keep or rescind the original violation ruling.

11.6 Beards and Excessive Facial Hair Policy

In order to comply with OSHA's standard on respiratory protection, beards and excessive facial hair will not be permitted on those personnel who must enter the Contaminant Reduction Zone (CRZ) or the Exclusion Zone (EZ).

SECTION 12
DECONTAMINATION PROCEDURES

12.1 General Considerations

Personnel involved with hazardous material handling may be exposed to compounds in a number of ways, despite the most stringent protective procedures. Personnel may come in contact with vapors, gases, mists, or particulates in the air, or may come in contact with Site media while performing work tasks. Use of monitoring instruments and equipment can also result in exposure to hazardous substances.

In general, personnel decontamination involves scrubbing with a non-phosphate soap/water solution followed by clean water rinses. All PPE disposable items will be disposed of in a dry container. Certain parts of contaminated respirators, such as harness assemblies and leather or cloth components, are difficult to decontaminate. If grossly contaminated, they may have to be discarded. Rubber components can be soaked in soap and water and scrubbed with a brush. In addition to being decontaminated, all respirators, non-disposable protective clothing, and other personal articles must be sanitized before they can be used again. The manufacturer's instructions should be followed in sanitizing the respirator masks. The Site Safety Officer will be responsible for supervision the proper protective equipment. Additional detail on decontamination can be found in Attachment D.

Avoidance of exposure to hazardous materials that may be at the Site shall be practiced at all times during site activities. Personnel performing investigative tasks should be conscious of their potential for exposure caused by fatigue or unsafe work practices. Great care should be taken when removing potentially contaminated PPE, or when handling potentially contaminated sampling equipment, sample containers, etc.

Smoking, eating, and drinking, or other activities which promote hand to mouth contact will not be permitted in the EZ or the CRZ. Beverages for use in the event of heat stress/fatigue shall be located in the Support Zone. If work sites are far from the SZ, contaminants are minimal, and the SSO authorizes, beverages for heat stress may be located in a designated clean area of the EZ. Proper decontamination procedures for personnel entering the clean area must be followed.

SECTION 13
EMERGENCY PROCEDURES CONTINGENCY PLAN

13.1 Emergency Contact Telephone Numbers

In the event of an emergency during implementation of field activities, the following telephone contact numbers shall be readily available to all field personnel. A copy of these numbers shall be posted conspicuously near the radio telephones in the field vehicles, and shall also be in possession of the SSO and sampling personnel:

Emergency Contacts	Person or Agency	Telephone
Ambulance		911
Police	Town of Smyrna Police	(302) 653-3483
Fire	Smyrna Fire Department	(302) 653-9858
Hospital	Kent General Hospital	(302) 734-4701
Clark Equip. Co. Contact	David Jones	(219) 239-0195
Metals Masters Contact	Rick Murphy	(302) 653-3000
ERM Project Manager	David Steele	(215) 524-3526
ERM H & S Coordinator	Robert Deist	(215) 524-3775
Medical Surveillance	Paoli Memorial Hospital	(215) 648-1000

13.2 Incident Reporting Procedures

Adherence to this site-specific Health and Safety Plan, and any additional safety rules and regulations, will significantly reduce the likelihood of personnel being exposed to toxic substances above permissible exposure limits. However, in the event an incident does occur, it is imperative that specific reporting procedures be followed so that appropriate corrective action can be taken by the ERM Health and Safety *Coordinator* (HSC) and the ERM Project Manager. Upon notification of an incident, the ERM HSC will contact the appropriate technical personnel for recommended medical diagnosis and, if necessary, treatment. The ERM Project Manager and the ERM H&S Manager will investigate facility/site conditions to determine: (1)

whether and at what levels the incident actually occurred, (2) the cause of such the incident, and (3) the means to prevent the incident from recurring.

An incident reporting form (Exhibit 13-1) has been developed so that consistent and appropriate information is obtained regarding employee exposures. The form will be completed by the ERM HSC and the exposed individual. The form will be filed with the employee's medical and safety records to serve as documentation of the incident and the actions taken.

13.3 Emergency Response Procedures

In the event of an emergency, the Site Safety Officer will assume responsibility for coordinating the response to all emergencies. The SSO responsibilities will also include:

1. Notification of appropriate individuals, authorities and/or health investigation.

2. Ensuring that the following safety equipment is available at all times at the Site: eyewash station, first aid supplies, and fire extinguishers.

3. Having a working knowledge of all safety equipment available at the site; and

4. Ensuring that a map which details the most direct route to the nearest hospital is prominently posted on-site, in addition to the emergency telephone numbers.

13.3.1 Accidents and Injuries

In the event of a safety or health emergency at the Site, appropriate emergency measures will immediately be taken to assist those who have been injured or exposed and to protect others from hazards. The Site Safety Officer will be immediately notified, and will respond according to the seriousness of the injury. Personnel trained in first-aid will be present at all times during site activities to provide appropriate treatment of injuries or illness incurred during operations. The ERM Project Manager will be immediately informed of any serious injuries. The project field personnel shall take the injured party and transport (if possible) to the nearest hospital for treatment, after determining whether personnel decontamination can be performed on the injured party. If a particular injury precludes the possibility of personnel decontamination, the SSO shall notify all emergency personnel of the potentially contaminated PPE and provide any assistance necessary in properly decontaminating or removing the PPE. In situations of minor injuries, an injured party may be transported to the nearest hospital. For this reason, the hospital route map shall be

EXHIBIT 13-1

Environmental Resources Management Inc.
855 Springdale Drive
Exton, PA 19341

ERM INCIDENT REPORT FORM

CLIENT NAME:_____ LOCATION OF INCIDENT:
DATE:_____

EMPLOYEE:_____ TYPE OF INCIDENT:_____

EMPLOYEE JOB TITLE:_____

SPECIFIC JOB AT TIME OF INCIDENT:_____

LEVEL OF PROTECTION WORN AT TIME OF EXPOSURE:

SUMMARY OF WHAT OCCURRED: _____

_____

_____

_____

_____

_____

_____

_____

_____

_____

ACTIONS TAKEN TO CORRECT SITUATION
(Engineering, PPE, etc.):_____

_____

_____

_____

_____

EMPLOYEE SIGNATURE:_____

SITE SAFETY OFFICER:_____

ERM H&S COORDINATOR:_____

TIME AND DATE OF REPORT:_____

The ERM Group

made available to all personnel. *The Hospital Route map is included on the following page.*

In the event of an environmental release, the field personnel shall make an initial effort to control or stop the spread of *the release*, if at all possible, without compromising personnel health and safety. The SSO should immediately contact the Delaware Emergency Control Center to report the release, and request the necessary assistance from the regulatory agency personnel to prevent health impact to surrounding residences and businesses.

In the event of potential fire, explosion, or other imminent hazard, the SSO shall initiate evacuation procedures using an appropriate warning device readily audible to all field personnel (i.e., walkie-talkie system, car horn, air horn, etc.) and will sound the device for a minimum of ten seconds. All personnel on-site will evacuate to the support zone, and will assist the SSO in controlling access to the Site once the emergency evacuation has been initiated. The SSO will assist the emergency response personnel, and document all activities occurring during an evacuation or emergency in the site logbook.

13.3.2 Directions to Nearest Hospital

Take Route 6 west (Smyrna-Clayton Avenue) to Route 13 which is approximately one mile. Take Route 13 south approximately 10 miles to South State Street. Turn right onto South State Street. Follow South State Street until reaching the Kent County Hospital located on the right hand side.

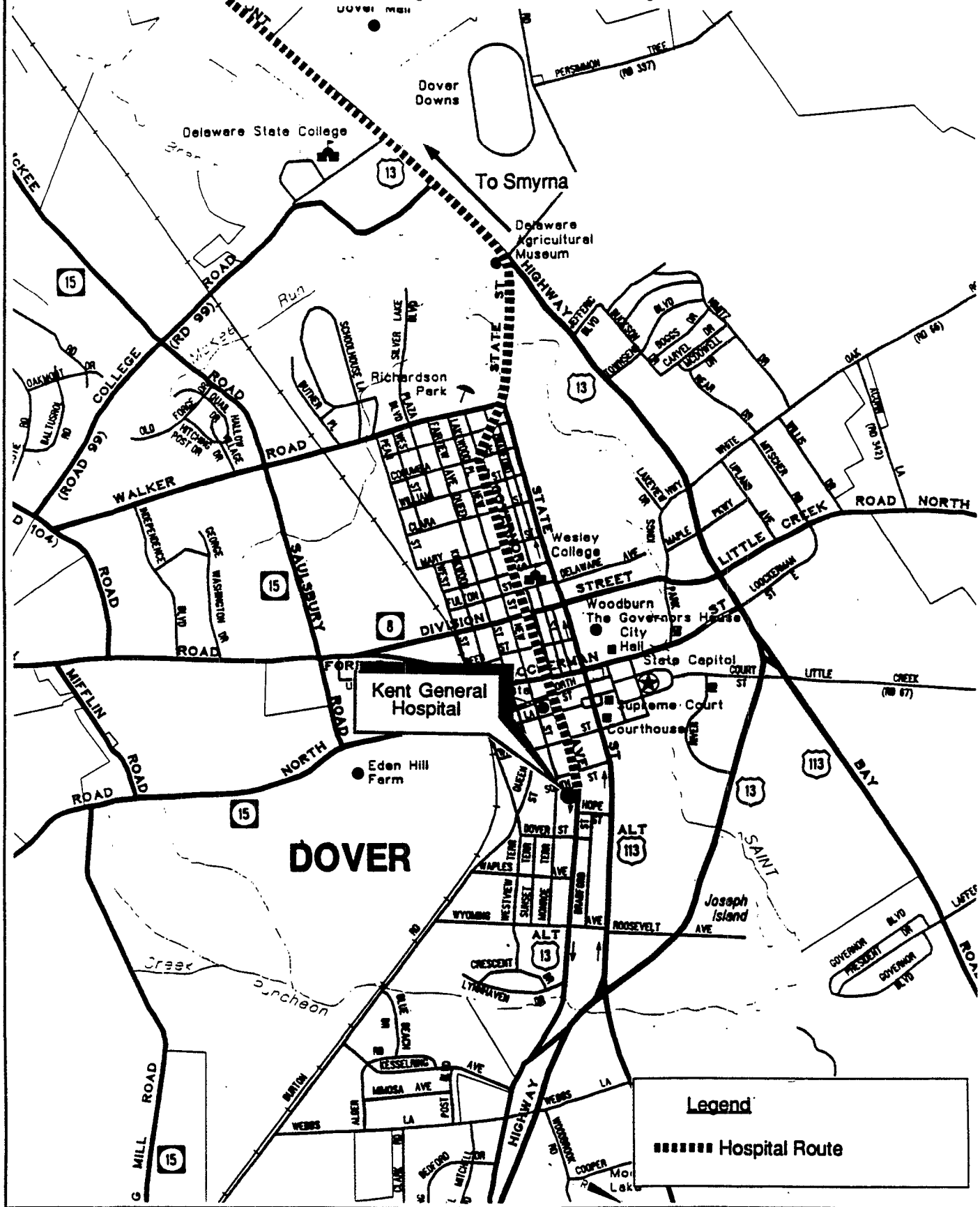
13.3.3 Overt Personnel Exposure

Generic first aid procedures, designed to address initial actions to be taken with the victim in the event of a personnel exposure, are briefly outlined below. Typical responses to overt personnel exposure include:

Skin/Eye Contact: Use copious amounts of soap and water, and flush skin for at least 15 minutes. Wash/rinse affected area thoroughly, then provide appropriate medical attention. Eyewash and drench system (water hoses) will be provided on site at the CRZ and/or Support Zone as appropriate. Eyes should be rinsed for 15 minutes after contact with any chemical contamination.

Inhalation Exposure: Move to fresh air immediately and/or, if necessary, decontaminated and transport to hospital.

Hospital Route Map



WO# C2701	Drawn By / Date: P. Kryven 11.11.91	Checked By / Date E. Sullivan:	The ERM Group
	Revised By / Date: P. Kryven 11.12.91	Checked By / Date: E. Sullivan 11.12.91	

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13.4 Additional Health and Safety Precautions and Procedures

Heat Stress or Cold Exposure

The timing of this project may be such that heat stress or cold exposure may pose a threat to the health and safety of site personnel. Work/rest regimens will be employed as necessary so that personnel do not suffer adverse effects from heat stress or cold exposure. Special clothing and an appropriate diet and fluid intake will be recommended to all site personnel to further reduce these temperature-related hazards. The work/rest regimens will be developed following the guidelines in the ACGIH, Threshold Limit Values and Biological Exposure Indices for 1987-1988. *These guidelines are applicable to the use of Tyvek protective clothing which is air permeable.* A complete heat stress program can be found in Attachment E.

Heavy Machinery/Equipment

All site employees must remain aware of those site activities that involve the use of heavy equipment and machinery. Respiratory protection and protective eyewear may be worn frequently during site activities. This protective equipment significantly reduces peripheral vision of the wearer. Therefore, it is essential that all employees at the Site exercise extreme caution during operation of equipment and machinery to avoid physical injury to themselves or others.

Construction Materials and Site Refuse

All construction materials and site refuse should be contained in appropriate areas or facilities. Site personnel should make certain that fencing, cement, etc. are not scattered throughout the area of activity and that all trash and scrap materials are immediately and properly disposed.

Additional Safety Practices

The following important safety precautions will be enforced during this investigation:

1. Eating, drinking, chewing gum, or tobacco, smoking, or any practice that increase the probability of hand-to-mouth transfer and ingestion of material is prohibited in any area designated as contaminated.

2. Hands and face must be thoroughly washed upon leaving the work area and before eating, drinking, or any other activity.

3. Whenever decontamination procedures for outer garments are in effect, the entire body should be thoroughly washed as soon as possible after the protective garment is removed.

4. No facial hair which interferes with the effectiveness of a respirator will be permitted on personnel required to wear respiratory protection equipment. The respirator must seal against the face so that the wearer receives air only through the air purifying cartridges attached to the respirator. Fit testing shall be performed prior to respirator use to ensure a proper seal is obtained by the wearer.

5. Contact with potentially contaminated surfaces should be avoided whenever possible. One should not walk through puddles, mud, or other discolored surfaces; kneel on ground; or lean, sit or place equipment on drums, containers, vehicles, or the ground.

6. Medicine and alcohol can potentiate the effect for exposure to certain compounds. Prescribed drugs and alcoholic beverages should not be consumed by personnel involved in field activities.

7. Personnel and equipment in the work areas should be minimized, consistent with effective site operations.

8. Work areas for various operational activities should be established.

9. Procedures for leaving the work area must be planned and implemented prior to going to the site. Work areas and decontamination procedures must be established on the basis of prevailing site conditions.

10. Respirators will be issued for the exclusive use of one worker and will be cleaned and disinfected after each use by the worker.

11. Safety gloves and boots shall be taped to the disposable, chemical-protective suits as necessary.

12. All unsafe equipment left unattended will be identified by a "DANGER, DO NOT OPERATE" tag.

13. Noise mufflers on equipment and/or hearing protection for personnel may be required for all work around heavy equipment. This requirement will be made at the discretion of the Site Safety Officer.

14. Cartridges for air-purifying respirators in use will be changed daily at a minimum.

15. Self-contained breathing apparatus (SCBA) and air-purifying respirators will be inspected daily by the Site Safety Officer.

16. All activities in the exclusion zone will be conducted using the "Buddy System". The Buddy is another worker fully dressed in the appropriate PPE, who can perform the following activities:

    - Provide his/her partner with assistance;
    - Observe his/her partner for signs of chemical or heat exposure;
    - Periodically check the integrity of his/her partner's PPE; and
    - Notify others if emergency help is needed.

ATTACHMENT A

PERSONNEL PROTECTIVE EQUIPMENT

A.1 Protective Equipment

All personnel must be provided with appropriate personal safety equipment and protective clothing. Each individual will be properly trained in the use of this safety equipment before the start of field activities. Safety equipment and protective clothing shall be used as directed by the Site Safety Officer. All such equipment and clothing will be cleaned and maintained in proper condition by project personnel. The Site Safety Officer will monitor the maintenance of personnel protective equipment to ensure proper procedures are followed.

Personal protective equipment will be worn at all times, as designated by the Health and Safety Plan. Levels of protective clothing and equipment have been assigned to specific work tasks.

The personal protective equipment levels designated below are in conformance with EPA criteria for Level B, C, and D protection. All respiratory protective equipment used will be approved by *National Institute of Occupational Safety and Health/Mine Safety and Health Administration* (NIOSH/MSHA).

A.2 Level B. Protection

A. Pressure demand cascade air-line system or other suitable self-contained, pressure demand breathing apparatus.

B. Chemical-resistant clothing such as Poly-coated Tyvek® or Saranex®. Suits will be one piece with booties and elastic wrist bands.

C. Outer nitrile and inner latex surgical gloves.

D. Leather boots with rubber overboots.

E. Water-resistant tape over protective clothing as necessary.

F. Options as required:

1. Coveralls
2. Disposable outer boots
3. Face shield

4. Escape mask

5. Hearing protection

A.3 Level C Protection

A. Full-face or half-face air purifying respirator equipped with appropriate organic vapor/dust canisters or cartridges.

B. Chemical-resistant clothing such as Tyvek®, Poly-coated Tyvek® or Saranex®. Suits will be one piece with hoods, booties and elastic wrist bands.

C. Outer nitrile gloves and inner latex surgical gloves.

D. Leather boots with rubber overboots.

E. Safety Glasses

F. Options as required:

1. Coveralls

2. Disposable outer boots

3. Escape mask

4. Face shield

5. Hearing protection

6. Water-resistant tape

A.4 Level D. Protection

A. Coveralls or long sleeve shirts and long pants.

B. Outer nitrile gloves at a minimum for all material handling activities. Inner latex surgical gloves are recommended where practical.

C. Leather boots with rubber overboots.

D. Level C protection readily available.

E. Safety Glasses

F. Options as required:

1. Disposable outer boots
2. Hard hat
3. Hearing protection
4. Chemical-resistant gloves

ATTACHMENT B

MEDICAL MONITORING

The Occupational Safety and Health Administration (OSHA) has established requirements for a medical surveillance program designed to monitor and reduce health risks for employees potentially exposed to hazardous materials (29 CFR 1910.120). This program has been designed to provide baseline medical data for each employee involved in hazardous waste operations including field activities, and to determine his/her ability to wear personal protective equipment, such as chemical resistant clothing and respirators. Employees who wear or may wear respiratory protection must be provided respirators as regulated by 29 CRF 1910.134. This Standard requires that an individual's ability to wear respiratory protection be medically certified before he/she performs designated duties. Where medical requirements of 29 CFR 1910.120 overlap those of 29 CFR 1910.134, the most stringent of the two will be enforced.

The medical examinations must be administered on a pre-employment and annual basis and as warranted by symptoms of exposure or specialized activities. These examinations shall be provided by employers without cost or loss of pay to the employee. For the purposes of this Health and Safety Plan, all subcontractors shall assume the employer's responsibility in obtaining the necessary medical monitoring and training for their employees pursuant to this section of 29 CFR 1910.120.

The examining physician is required to make a report to the employer of any medical condition which would place such employees at increased risk of wearing a respirator or other personal protective equipment. Each employer engaged in site work shall assume the responsibility of maintaining site personnel medical records as regulated by 29 CFR 1910.120 where applicable. Exemption from the medical surveillance program may be allowed by the ERM Manger of Safety and Health or his designee. These exemptions will be based on his interpretation of the requirements of 1910.120 relative to each individual exemption request.

All employees contracted to work at the site designated by this Plan will be responsible to insure their employees have received the proper medical tests as regulated by 29 CFR 1910.120 and shall provide the contractor with certification of same.

ATTACHMENT C

PERSONNEL TRAINING

Site personnel associated with those field activities in which the potential for exposure to hazardous substances above the PEL exists will be required to participate in a health and safety training program that complies with criteria set forth by ERM and OSHA as per interim final regulation 29 CFR 1910.120. This program will instruct employees on general health and safety principles and procedures, proper operation of monitoring instruments, and use of personal protective equipment.

In addition, site employees will undergo site-specific training prior to the start-up of any given project or task. As activities change at a particular site, related training will address potential hazards and associated risks, site operating procedures, emergency response and site control methods to be employed.

Specialized training will be provided as dictated by the nature of site activities. Specialized training will be provided for activities such as confined space entry, excavations and handling of unidentified substances. Employees involved in these types of activities will be given off-site instruction regarding the potential hazards involved with safety activities and the appropriate health and safety procedures to be followed. Off-site instruction is meant to include any area where employees will not be exposed to site hazards.

Site personnel involved in the field activities will have received the appropriate basic training plus additional specific training where needed. This Health and Safety Plan must be distributed to all subcontractors prior to the start of field activities. A pre-operation meeting will be held to discuss the contents of the Plan. Specialty training will be provided as determined by task and responsibility. All training of personnel will be conducted under direct supervision of a trained Health and Safety Officer.

Exemptions from training may be approved by the ERM Manager of Health and Safety or his designee.

ATTACHMENT D

DECONTAMINATION

D.1 General

Personnel involved with hazardous material handling may be exposed to compounds in a number of ways, despite the most stringent protective procedures. Personnel may come in contact with vapors, gases, mists, or particulates in the air, or may come in contact with site media while performing work tasks. Use of monitoring instruments and equipment can also result in exposure to hazardous substances.

In general, decontamination involves scrubbing with a non-phosphate soap/water solution followed by clean water rinses. All disposable items will be disposed of in a dry container. Certain parts of contaminated respirators, such as harness assemblies and leather or cloth components, are different to decontaminate. If grossly contaminated, they may have to be discarded. Rubber components can be soaked in soap and water and scrubbed with a brush. In addition to being decontaminated, all respirators, non-disposable protective clothing, and other personal articles must be sanitized before they can be used again unless they are assigned to individuals. The manufacturer's instructions should be followed in sanitizing the respirator masks. The Site Safety Officer will be responsible for supervising the proper protective equipment.

D.2 Standard PPE Decontamination

The Site Safety Officer will monitor decontamination procedures to ensure their effectiveness. Modifications of the decontamination procedure may be necessary as determined by the Site Safety Officer's observations.

Level B - Personal Protection Decontamination Procedure

Step 1 -- Segregated Equipment Drop

Proceed to the Contaminant Reduction Zone and deposit equipment (tools, sampling devices, notes, monitoring instruments, radios, etc) used *in the Exclusion Zone* onto plastic drop cloths.

Step 2 -- Boot Covers and Glove Wash

Outer Boot covers and outer gloves should be scrubbed with a decontamination solution of detergent and water.

Step 3 -- Rinse Off Boot Covers and Gloves

Decontamination solution should be rinsed off boot covers and gloves using generous amounts of water. Repeat as many times as necessary.

Step 4 -- Tape Removal

Remove tape from around boots and gloves and place into container with plastic liner.

Step 5 -- Boot Cover Removal

Remove disposable boot covers and place into container with plastic liner.

Step 6 -- Outer Glove Removal

Remove outer gloves and deposit in container with plastic liner.

Step 7 -- Suit/Safety Boot Wash

Completely wash splash suit, SCBA, gloves, and safety boots. Care should be exercised that no water is allowed into the SCBA regulator. It is suggested that SCBA regulator be wrapped in plastic.

Step 8 -- Suit/Safety Boot Rinse

Thoroughly rinse off all decontamination solution from protective clothing.

Step 9 -- Tank Changes

This is the last step in the decontamination procedure for those workers wishing to change air tanks and return to the exclusion zone. The worker's air tank is exchanged, new outer glove and boot covers are donned, and joints taped.

Step 10 -- Removal of Safety Boots

Remove safety boots and deposit in container with a plastic liner.

Step 11 -- SCBA Backpack Removal

Without removing face piece, remove the SCBA backpack and place it on a table. Then disconnect the face piece from the remaining SCBA unit and proceed to the next station.

Step 12 -- Splash Suit Removal

With care, remove splash suit. The exterior of the splash suit should not come in contact with any inner layers of clothing.

Step 13 -- Inner Glove Wash

The inner gloves should be washed with a mild decontamination solution (detergent/water).

Step 14 -- Inner Glove Rinse

Generously rinse inner gloves with water.

Step 15 -- Face Piece Removal

Without touching face with gloves, remove face piece. Deposit face piece into a container which has a plastic liner.

Step 16 -- Inner Glove Removal

Remove inner glove and deposit in container with plastic liner.

Step 17 -- Field Wash

Wash hands and face thoroughly. If highly toxic, skin corrosive, or skin-absorbent materials are known or suspected to be present, take a shower.

Level C and Level D Personal Protective Decontamination

The decontamination procedure for Level C and Level D personal protection will employ applicable steps detailed in the Level B decontamination process.

ATTACHMENT E
HEAT STRESS GUIDELINES

The following should be used as guidelines in controlling heat stress. The Site Health and Safety Officer has the responsibility to monitor heat stress throughout each day and to make work/rest recommendations as appropriate. All workers are expected to follow the work/rest cycles.

Heat stress decisions will be based mostly on physiological measurements (pulse rate, skin temperature) and environmental measurements by the Wet Bulb Globe Temperature (WBGT) monitors. Additional environmental data will also be recorded daily and considered in heat stress evaluations.

Initially, work/rest cycles will be established using pulse rates and the following guidelines. This work/rest schedule may be modified at the discretion of the Health and Safety Officer. The WBGT readings in this table are actual readings - no additional factors should be added:

WORK/REST SCHEDULE

WBGT (°C)		*(Minutes)*
LEVEL C&B	LEVEL D	WORKING-RESTING
<22.5	<24	NORMAL
22.5-24.4	24-25.9	60-15
24.5-26.4	26-27.9	45-15
26.5-29.4	28-30.9	30-30
29.5-30.4	31-31.9	15-45
30.5-32	32-33.5	15-60
>32	>33.5	CEASE WORK

Daily Protocol

- WBGT Readings will be taken:
  - at the beginning of the work day
  - mid-morning
  - noon
  - mid-afternoon
  - at the end of the work day

- WBGT readings will be taken at all major work areas and at outside rest stations.

- Employee body weights (semi-nude) should be taken immediately before work and at the end of the work day. If this procedure is not practical for the worksite encourage workers to weigh themselves at home. If the weight loss exceeds 1.5%, the worker should drink more liquids during that evening and the following work days. The worker should also be monitored during the next few work days to insure the weight loss does not continue at an unacceptable rate.

- Pulse rates must be monitored routinely throughout the workday, frequency depending upon WBGT readings. At minimum, the most active member of each work crew should be monitored during the first two breaks in the morning and the first break after lunch.

- Pulse rates will be taken as follows:

    - at the end of a cycle of work, the worker goes to a nearby location and sits on a stool or straight chair. At the moment he is seated the observer starts a stopwatch. At 30 seconds the observer begins a pulse count, having previously palpated the radial pulse. This count is continued until one minute. The 30-second count is multiplied by 2 and recorded as "P_1"

    - if P_1 exceeds 120, an additional pulse will be taken starting at 2 minutes, 30 seconds to 3 minutes; multiplied by 2 and recorded as P_3.

- Pulse rates readings: (Apply to workers wearing both permeable and impermeable clothing.)

  120 and below (P_1) - Worker will be allowed to continue the scheduled work/rest cycle.

  Exceeding 120 (P_1) - Worker will remain in the rest area until pulse rate returns to 90, or below; additional monitoring will depend upon the pulse rate recovery.

- Pulse rate recovery - for individual with P_1 greater than 120.

Patterns	P_3	P_1-P_3
Satisfactory (S)	<90	-
High (H)	≥90	≥10
No recovery (N)	≥90	<10

- Satisfactory patterns need no further comment.

  - High recovery patterns indicate work at a high metabolic level with little or no accumulated body heat. Individuals showing this condition should be monitored during the next breaks while work periods are reduced until P_1 is 120 or below.

  - "No recovery" patterns indicate too much personal stress. Individuals showing "no recovery" heart rate patterns return to a shaded or air conditioned rest area and rest for a period no less than one hour. Site Health and Safety Officer must monitor the workers and determine if additional medical assistance is needed.

- Oral temperature readings will be taken if other signs of heat stress are apparent.

- Workers will also be visually monitored for signs of heat stress. The signs of heat exhaustion include flushed face, sweating, dizziness, and nausea. The signs of heat stroke include pale, dry hot skin and no evidence of sweating.

- Fluid intake should be encouraged for workers throughout the day. Workers should frequently drink small amounts; the equivalent of one cup every 15-20 minutes. Fluids that replace electrolytes (such as Gatorade) should also be available. Workers should eat well balanced diets over the duration of the project. Workers should also be encouraged to salt their food lightly if they do not typically use salt with their meals.

- Acclimatization to heat involves a series of physiological and psychological adjustments that occur in an individual during the first week of exposure to hot environments. For their reason, the following work schedule applies for workers new to the site when conditions are such that controlled work/rest cycles are being used:

		Suggested Maximum Work
Day 1	-	2 hours
Day 2	-	3 hours
Day 3	-	4 hours
Day 4	-	6 hours
Day 5	-	8 hours

Deviations from this schedule may be done based on evaluations by the Site Health and Safety Officer.